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PIC ANALYSIS METHODS

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The report gives results of method evaluations for products of incomplete combustion (PICs): 36 proposed PICs were evaluated by previously developed gas chromatography/flame ionization detection (GC/FID) and gas chromatography/mass spectroscopy (GC/MS) methods. It also gives results of method evaluations of 59 additional principal organic hazardous constituents (POHCs) from the amended list of compounds from Appendix VIII of 40 CFR, Part 261. The report contains chromatograms, bar-graph mass spectra, and calibration curves for each compound. Relative retention times, detection limits, precision data, and mass spectra are tabulated for each compound. The report also reviews recent literature on the formation of PICs.

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Incinerators	Gas Chromatography	Stationary Sources		07D
Wastes	Mass Spectroscopy	Hazardous Waste	14G	
Toxicity	Flames	Products of Incomplete Combustion (PICs)	06T	
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FOREWORD

This report has been prepared by Southern Research Institute as part of ongoing studies in support of regulatory programs of the Environmental Protection Agency's (EPA's) Air and Energy Engineering Research Laboratory at Research Triangle Park, North Carolina. This report contains criteria for the analysis methods for determination of products of incomplete combustion (PICs) and principal organic hazardous constituents (POHCs) from the combustion of hazardous waste. The data in this report are intended as a reference to be used by laboratories that are monitoring the hazardous-waste streams of incineration facilities. Because of subtle differences among laboratories and analytical instrumentations, analysts who participate in similar studies should generate data that will establish their own criteria for the analysis of PICs and POHCs.

ABSTRACT

The purpose of Work Assignment 004, Amendment 01 and Work Assignment 011 under Contract 68-02-3696 was to support the U.S. Environmental Protection Agency in its effort to license hazardous-waste incinerators.

A major part of these tasks was devoted to method evaluations for products of incomplete combustion. After examination of the literature, and consultation with the EPA project officer, personnel at LSU, EPA's Combustion Research Facility in Pine Bluff, AK, Arthur D. Little, Inc., EPA/HWERL's Center Hill Facility in Cincinnati, OH, and AEERL, 36 proposed PICs were evaluated by the previously developed GC/FID and GC/MS methods.

A second part of these tasks was devoted to method evaluation of 59 additional POHCs from the amended list of compounds from Appendix VIII 40 CFR part 261.

This report contains chromatograms, bar-graph mass spectra and calibration curves for each compound. Relative retention times, detection limits, precision data and mass spectra are tabulated for each compound. The report also includes a review of recent literature on the formation of PICs.

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(D) phosphorodithioic acid, *S*,*S*'-methylene *O*,*O*,*O*',*O*'-tetraethyl ester, and
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(D) phosphorodithioic acid, *O*,*O*-dimethyl ester, *S*-ester
with *N*-(mercaptomethyl)phthalimide,
(E) phosphonothioic acid, phenyl-, *O*-(4-bromo-2,5-dichloro-
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(B) phosphoric acid, 1,2-dibromo-2,2-dichloroethyl dimethyl ester,
(C) phosphorothioic acid, *O*,*O*-dimethyl-, *O*-(4-methylthio)-*m*-tolyl) ester, and
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(F) benzonitrile, 3,5-dibromo-4-hydroxy-,
(G) aniline, p-(phenylazo)-,
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(B) succinic acid, mercapto-, diethyl ester, *S*-ester with *O,O*-dimethyl phosphorodithioate,
(C) crotonic acid, 3-hydroxy-, *alpha*-methylbenzyl ester, dimethyl phosphate, (*E*)-,
(D) phosphorodithioic acid, *S,S'*-methylene *O,O,O',O'*-tetraethyl ester, and
(E) phosphorodithioic acid, *O,O*-dimethyl ester, *S*-ester with 3-(mercaptomethyl)-1,2,3-benzotriazin-4(3*H*)-one. 448

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ABBREVIATIONS AND SYMBOLS

amu	Atomic Mass Unit
Appendix VIII	Hazardous Constituents List (Part 261, 40 CFR)
CB	Chlorobenzenes
CDD	Chlorinated Dibenzo-*p*-dioxin
CDF	Chlorinated Dibenzofuran
CFR	Code of Federal Regulations
CHC	Chlorinated Hydrocarbons
CP	Chlorophenols
DFTPP	Decafluorotriphenylphosphine
DRE	Destruction and Removal Efficiency
EI	Electron Impact Ionization Mode
EPA	U.S. Environmental Protection Agency
g	Gram
GC	Gas Chromatography
GC/FID	Gas Chromatography/Flame-Ionization Detection
GC/MS	Gas Chromatography/Mass Spectrometry
h	Hour
HPLC	High-Performance Liquid Chromatography
HPLC/UV	High-Performance Liquid Chromatography/ Ultraviolet Spectrometry Detection
ID	Internal Diameter
is	Internal Standard
μg	Microgram (10^{-6} g)
μL	Microliter (10^{-6} L)
mg	Milligram (10^{-3} g)
mL	Milliliter (10^{-3} L)
mm	Millimeter (10^{-3} m)
MM5	Modified Method 5 Sampling Train
mol wt	Molecular Weight
ms	Millisecond
m/z	Mass to Charge Ratio
ng	Nanogram (10^{-9} g)
nm	Nanometer (10^{-9} m)
PAH	Polynuclear Aromatic Hydrocarbons
PCB	Polychlorinated Biphenyls
PCN	Polychlorinated Naphthalenes
PCDPE	Polychlorinated Diphenyl Ethers
PCS	Polychlorostyrenes
POHC	Principal Organic Hazardous Constituent(s)
PIC	Product(s) of Incomplete Combustion

(continued)

ABBREVIATIONS AND SYMBOLS
(continued)

QA	Quality Assurance
QC	Quality Control
RCRA	Resource Conservation and Recovery Act
RRF	Relative Response Factor
RSD	Relative Standard Deviation
SASS	Source Assessment Sampling System
SIM	Selected-Ion Monitoring
s	Second
SD	Standard Deviation
SoRI	Southern Research Institute
t_R	Retention Time
UV	Ultraviolet Spectroscopy

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SECTION 1

INTRODUCTION

Products of incomplete combustion (PICs) may be formed during the incineration of hazardous wastes. PICs may be defined as compounds in the stack gas of an incinerator but not present in the waste feed at a concentration of 100 µg/g or more (1). In some instances, the PICs formed may also be constituents of the waste feed, thus confusing the analysis of principal organic hazardous constituents (POHCs). The total amount of hazardous PICs can easily exceed the total of the original POHCs chosen to monitor the trial burn. The compounds may be identifiable as fragments of incineration feed constituents, products of complex recombinations, substitution reactions in the flame or post-flame zone, or compounds that enter the incineration process through other sources.

We have concentrated on applying previously developed POHC general analysis methods to the analysis of PICs. A large body of the literature on PICs is based on the findings of polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) in incinerator effluents. We have evaluated generalized GC/FID and GC/MS analysis methods for PCDDs and PCDFs. The methods are ones we previously developed, and will be used to analyze for specific POHCs as well as for screening studies. We wished to define how well the methods would work for dioxins and furans if one simply "looked for them" during an analysis that might be primarily for another compound. If one were to choose a method for only dioxins or furans, then one of the methods previously reported by Harless, Tiernan or others would probably be the method of choice (especially if the detection limit needed to be pushed as low as possible).

An amendment to Appendix VIII, Part 261, 40 CFR, has added 121 new organic compounds to the list of candidate POHCs. We have extended the generalized GC/FID and GC/MS procedures to many of these compounds.

The following specific tasks were undertaken to supplement the development of generalized test procedures and to assist those working in the area of incineration or combustion of hazardous waste:

- The evaluation of generalized analysis methods for 10 PCDDs and 3 PCDFs by GC/FID and GC/MS
- The evaluation of generalized analysis methods for 23 non-dioxin proposed PICs
- The evaluation of a GC/MS/SIM analysis method for 7 PCDDs and 2 PCDFs
- The evaluation of generalized analysis methods for 56 POHCs by GC/FID and 58 POHCs by GC/MS from the amendment to Appendix VIII, Part 261, 40 CFR (2)

- Consultation took place during these work assignments with personnel at LSU; at EPA's Combustion Research Facility in Pine Bluff, Arkansas; at Arthur D. Little, Inc.; and at EPA/HWERL's Facility in Center Hill, Cincinnati, Ohio.

The subsequent sections of this report describe the efforts of Southern Research Institute to evaluate and, when necessary, modify GC/FID and GC/MS generalized analysis procedures for PICs and POHCs. A brief literature survey of PIC formation and analysis is presented in Section 4. The technical approach and results and discussion of PIC and POHC analysis are in Sections 5 and 6.

SECTION 2

CONCLUSIONS

Previously developed generalized GC/FID and GC/MS methods were successfully applied to the determination of candidate PICs. A selected-ion monitoring GC/MS procedure for PCDDs and PCDFs was also evaluated. Generalized GC/FID and GC/MS procedures were extracted to POHCs from the amended Appendix VIII, Part 261, 40 CFR (2).

The generalized methods are suitable for the determination of PCDDs and PCDFs at or above the detection limits determined. For lower limits of detection, or more specificity, methods such as those previously developed by Tiernan (18) may be necessary. The methods are also applicable to substituted polycyclic aromatic hydrocarbons (PAHs).

The methods were standardized with selected organic compounds over concentration ranges of interest and showed acceptable precision in the determination of most of the compounds.

SECTION 3

RECOMMENDATIONS

Several recommendations for future investigations are as follows:

- The general analysis methods should be applied to PICs not yet evaluated.
- Other contractors, EPA personnel, and other experts should be conferred with to continue to identify PICs of interest.
- Based on our findings, the proposed screening program should be evaluated when applied to incinerator-effluent samples, extracts, and sorbent media.
- Aid should continue to be rendered to other contractors, incinerator operators, and EPA personnel in the application of the general analysis procedures.
- This large data base should be committed to computer files to assist those who have need for these data.

SECTION 4

FORMATION AND ANALYSIS OF PICs

During the incineration of hazardous compounds, other compounds not identified in the wastes may be formed. These compounds are known as products of incomplete combustion (PICs). The aim of incineration is to destroy all POHCs and to make sure that harmful levels of PICs, which may be formed during combustion, do not escape into the environment. Mr. George Huffman of EPA-Cincinnati recently defined PICs as organic compounds in the stack gas of an incinerator but not present in the waste feed at a concentration of 100 µg/g or higher (1). At the same conference Mr. Andrew Trenholm of Midwest Research Institute (MRI) stated that the total amount of hazardous PICs can easily exceed the total of the original POHCs. Compounds which are detected as PICs may be present because of one of three circumstances:

1. The compounds were originally present in the waste, but are below the ≈100-µg/g detection limit, and then are detected in the stack (low DRE).

2. The compounds were introduced from some source other than waste feed (scrubbing liquid used in control devices, inleak of ambient air through ductwork, or auxiliary fuel streams).

3. The compounds were occurring as actual combustion by-products.

Each of these circumstances probably contributes a certain percentage to the total amount of PICs present.

Trenholm, Hathaway, and Oberacker (3) have published a report describing how they collected samples at operating hazardous-waste-incineration facilities and analyzed the collected samples for PICs. Eight incinerator sites were sampled, and 29 compounds were classified as PICs. These compounds are listed in Table 1.

The 29 PICs listed in Table 1 are categorized in the MRI study into three different groups of reaction pathways. The first group would be made up of identifiable fragments of feed constituents which result from partial oxidation or simple substitution reactions. Several of the plants MRI studied exhibited PICs which were unique to the type of waste which was burned and were identifiable products of reactions of waste constituents. For example, two facilities which burned large quantities of aniline had high concentrations of nitrophenols in the stack.

The second group contained products which are the result of complex recombination or substitution reactions. This group would contain high-molecular-weight compounds such as naphthalene, fluoranthene, and pyrene.

TABLE 1. PICs FOUND IN STACK EFFLUENTS[a]

PIC	Number of sites	Concentrations, ng/L
Benzene	6	12 - 670
Chloroform	5	1 - 1330
Bromodichloromethane	4	3 - 32
Dibromochloromethane	4	1 - 12
Naphthalene	3	5 - 100
Bromoform	3	0.2 - 24
Chlorobenzene	3	1 - 10
Tetrachloroethylene	3	0.1 - 2.5
1,1,1-Trichloroethane	3	0.1 - 1.5
Toluene	2	2 - 75
o-Nitrophenol	2	25 - 50
Methylene chloride	2	2 - 27
Phenol	2	4 - 22
2,4,6-Trichlorophenol	1	110
Carbon disulfide	1	32
o-Chlorophenol	1	2 - 22
2,4-Dimethyl phenol	1	1 - 21
Methylene bromide	1	18
Bromochloromethane	1	14
Trichlorobenzene	1	7
Hexachlorobenzene	1	7
Diethyl phthalate	1	7
Pentachlorophenol	1	6
Dichlorobenzene	1	2 - 4
Chloromethane	1	3
Methyl ethyl ketone	1	3
Bromomethane	1	1
Pyrene	1	1
Fluoranthene	1	1

[a] Taken from Trenholm, A.; Hathaway, R.; Oberacker, D. Products of incomplete combustion from hazardous waste incinerators; Report, EPA-600/9-84-022. Incineration and treatment of hazardous waste--proceedings of the tenth annual research symposium.

The third group consisted of simple fragments which cannot be readily traced to any single reactant, but rather seem to be universal by-products of the combustion of organic compounds. This group was the most broadly represented among the plants studied and included chlorinated methanes, ethanes, and ethylenes. Bromoform was detected in large concentrations at one facility that was burning brominated wastes. Other PICs which could result from combustion of aromatics include benzene, phenol, toluene and chlorobenzene.

The combustion of chlorinated hydrocarbons (CHCs) has been shown to form PICs (4). In CHC combustion, pyrolysis of the parent molecules forms dechlorinated intermediates because of the weak C-Cl bond. The dechlorinated intermediates then undergo a variety of reactions: recombination to form other CHCs, further pyrolysis, oxidization, or polymerization.

As the chlorine content of the CHCs increases, the formation of soot is favored because of the weak C-Cl bond strength. Soot formation is undesirable because it is often related to the production of toxic compounds such as PAHs. The combustion of CHCs has also been reported to produce chlorinated dioxins, most notably 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) (5,6).

PCDDs and PCDF have been found in incinerator effluents. Olie and coworkers were the first to identify chlorinated dioxins in fly ash (7). They obtained fly-ash and flue-gas samples from the Netherlands and have since analyzed other fly ashes and found PCDDs and PCDFs in a method-comparison study (8).

Buser and co-workers observed more than 30 of the possible 75 PCDD isomers in fly-ash samples from a municipal incinerator and an industrial heating facility in Switzerland (9). Karasek and co-workers analyzed fly-ash samples from Japan, Canada, and the Netherlands and found tetra- through octa-CDD in all samples (10-15). The sum of the tetra-CDDs was 2 to 10 ng/g. Bumb and coworkers observed chlorodioxins present in particulate matter from various combustion sources and in dust and soil in the vicinity of combustion sources (5).

Emissions from incineration of raw chemical wastes from burns conducted on M/T Vulcanus were analyzed for TCDD and TCDF (16). There were no significant quantities of TCDD formed (detection limit 10.0 ppb); however, TCDF isomers (0.2 to 2 ng/m^3) were detected in the stack gas.

Representative stack and fly-ash samples were analyzed from various combustion sources by Chiu et al. (17). Sample types included a large modern coal-fired thermal generating station, a 30-year-old coal boiler, a large modern heat-recovery incinerator, and a small, modular controlled-air incinerator. Additional samples included fly ash from a low-temperature incinerator, off-gases sampled during the burning of pentachlorophenol-impregnated boxes, and samples from the plasma pyrolysis of PCBs. PCDDs and PCDFs were in samples from all combustion sites investigated with the exception of a coal-fired central heating plant.

An incinerator used to heat a steam boiler designed for energy recovery from the combustion of municipal refuse was sampled and the effluents analyzed by Tiernan et al. (18). The effluents contained many chlorinated compounds. The concentrations of the classes of compounds were on the order of chlorophenols (CPs) > chlorobenzenes (CBs) > PCDFs > PCDDs > PCBs. CPs and CBs were the major combustion products, while the levels of PCBs were essentially negligible. The large number of TCDD isomers found were thought to result from the high level of CPs and CB compounds in the combustion environment.

Karasek and co-workers (19) have recently reported a complete analysis of fly-ash extracts from municipal incinerators in Ontario, Canada, and Oslo, Norway. They identified more than 200 organic components in the extracts. The fly-ash extracts were first fractionated into functional-group classes by a gradient-elution normal-phase HPLC procedure. These fractions were then analyzed by capillary GC and capillary GC/MS. Compound identification was based on matching observed mass-spectra with those of injected standards and from reference mass-spectral libraries. The retention indices of components in samples were also used to facilitate identification. Compounds found included hydrocarbons, phthalate esters, PAHs and polychlorinated organic compounds. PCDDs and PCDFs were present exclusively in one fraction. However, standards for many of the PCDD and PCDF isomers were not available, and thus it was not possible to quantify these isomers individually.

The compounds identified in the fly-ash extracts are listed in Tables 2 through 4.

The first fraction contained aliphatics, n-alkanes and alkenes, covering a wide range of boiling points. Fraction 2 contained PAHs with two to three rings, sulphur-containing PAHs, chlorinated oxy-PAHs with one to three rings, PCDDs, and PCDFs. Fraction 3 contained nitrogen-containing PAHs, oxygenated PAHs, chlorinated oxy-PAHs, and PAHs with three or more rings. Fraction 4 contained a large number of compounds which could not be identified because of a lack of standards. Fraction 5 contained compounds attributed to impurities in the system (e.g., butyl benzyl phthalate and dioctyl phthalate). Table 5 shows the levels of PCDDs determined in the two fly ashes.

In the research cited above (6-18) and in other work (19-20), PCDDs, PCDFs, CBs, PCBs, polychloronaphthalenes (PCNs), and PAHs were found in fly-ash samples from municipal incinerators. One report (5) indicates that PCDDs are formed in pyrolytic and combustion processes other than the incineration of municipal wastes. And PCDDs were found on particulate matter from fossil-fueled powerhouses, gasoline- and diesel-powered automobiles and trucks, fireplaces, charcoal grills, and cigarettes. Lustenhouwer et al. (20) and Cavallaro et al. (21) made some general hypotheses on the formation of chlorinated compounds in the pyrolytic processes.

PCDDs and PCDFs are present in the refuse in trace amounts and are not always effectively destroyed. It is possible that PCDDs and PCDFs could enter the refuse through herbicide formulations, treated wood, or products containing polychlorinated biphenyls. However, whenever PCDDs or PCDFs are found in compost, they are present at a much lower level than is found in the fly ash.

TABLE 2. COMPOUNDS IDENTIFIED IN HPLC FRACTION 2 OF ONTARIO FLY-ASH EXTRACT[a]

Individual isomers of some compounds are not identifiable by techniques used in this procedure.

No.	Compound	Mol wt
1	Tetramethylbenzene isomer	134
2	Tetramethylbenzene isomer	134
3	Trichlorobenzene isomer	180
4	Naphthalene	128
5	Trichlorobenzene isomer	180
6	2-Methylnaphthalene	142
7	1-Methylnaphthalene	142
8	Tetrachlorobenzene isomer	214
9	Biphenyl	154
10	1-Ethylnaphthalene	156
11	2,6- or 2,7-Dimethylnaphthalene	156
12	1,3- or 1,7- or 1,6-Dimethylnaphthalene	156
13	2,3- or 1,4- or 1,5-Dimethylnaphthalene	156
14	Acenaphthylene	152
15	Methylbiphenyl	168
16	3- or 4-Methylbiphenyl	168
17	Dibenzofuran	168
18	Pentachlorobenzene	248
19	2,3,6-Trimethylnaphthalene	170
20	Nonylbenzene	204
21	2,4,6-Trichlorophenol	196
22	Unidentified	182
23	Bromotetrachlorobenzene	292
24	Decylbenzene	218
25	Hexachlorobenzene	282
26	1- or 2-Methylfluorene	180
27	Bromodichloromethylphenol	254
28	Dibenzothiophene	184
29	2,3,4,6-Tetrachlorophenol	230
30	Phenanthrene	178
31	Tetrachloroacenaphthylene isomer	288
32	Unidentified compound with 6 chlorines	306
33	Tetrachloroacenaphthylene isomer	288
34	Dihydropyrene or dihydrofluoranthene	204
35	Dichlorodibenzofuran	236
36	3-Methylphenanthrene	192
37	Tetrachlorobenzene dicarbonitrile isomer	264
38	2-Methylanthracene	192
39	1-Methylanthracene (or 1-, 4-, or 9-methylphenanthrene)	192

(continued)

TABLE 2 (continued)

No.	Compound	Mol wt
40	Tetrachlorobenzene dicarbonitrile isomer	264
41	2,3-Dichlorodibenzodioxin	252
42	Phenylnaphthalene	204
43	Chlorophenylethynylbenzene	212
44	2-Ethylphenanthrene	206
45	Trichlorodibenzofuran isomer	270
46	Trichlorodibenzofuran isomer	270
47	Fluoranthene	202
48	Trichlorodibenzofuran isomer	270
49	Trichlorodibenzofuraon isomer	270
50	Pentachloronaphthalene isomer	298
51	Trichlorodibenzodioxin isomer	286
52	Pyrene	202
53	Trichlorodibenzofuran isomer	286
54	Pentachloronaphthalene isomer	298
55	Trichlorodibenzodioxin isomer	286
56	Pentachloronaphthalene isomer	298
57	Tetrachlorobiphenyl isomer	290
58	Tetrachlorodibenzofuran isomer	304
59	Tetrachlorodibenzofuran isomer	304
60	Tetrachlorodibenzodioxin isomer	320
61	Tetrachlorodibenzodioxin isomer	320
62	Tetrachlorodibenzofuran isomer	304
63	Tetrachlorodibenzodioxin isomer	320
64	Hexachloronaphthalene isomer	332
65	1,2,3,4-Tetrachlorodibenzodioxin	320
66	Tetrachlorodibenzodioxin isomer	320
67	Tetrachlorodibenzodioxin isomer	320
68	Benzo[ghi]fluoranthene	226
69	Pentachlorodibenzofuran isomer	338
70	Pentachlorodibenzofuran isomer	338
71	Pentachlorodibenzodioxin isomer	354
72	Pentachlorodibenzofuran isomer	338
73	Pentachlorodibenzodioxin isomer	354
74	Pentachlorodibenzodioxin isomer	354
75	Pentachlorodibenzodioxin isomer	354
76	Pentachlorodibenzodioxin isomer	354
77	Pentachlorodibenzodioxin isomer	354
78	Pentachlorodibenzodioxin isomer	354
79	Hexachlorodibenzofuran isomer	372
80	Hexachlorodibenzofuran isomer	372
81	Hexachlorodibenzodioxin isomer	388
82	Hexachlorodibenzodioxin isomer	388

(continued)

TABLE 2 (continued)

No.	Compound	Mol wt
83	Hexachlorodibenzodioxin isomer	388
84	Hexachlorodibenzofuran isomer	372
85	1,2,3,4,7,8-Hexachlorodibenzodioxin	388
86	Hexachlorodibenzodioxin isomer	388
87	Nonachlorobiphenyl isomer	460
88	Decachlorobiphenyl	494
89	Heptachlorodibenzofuran isomer	406
90	1,2,3,4,6,7,9-Heptachlorodibenzodioxin	422
91	1,2,3,4,6,7,8-Heptachlorodibenzodioxin	422
92	Octachlorodibenzodioxin	456

[a] Taken from Tong, H.Y.; Shore, D.L.; Karasek, F.W.; Helland, P.; Jellum, E. Identification of organic compounds obtained from incineration of municipal waste by high-performance liquid chromatographic fractionation and gas chromatography/mass spectrometry. J. Chromatogr. 285: 423-441; 1984.

TABLE 3. COMPOUNDS IDENTIFIED IN HPLC FRACTION 3 OF ONTARIO FLY-ASH EXTRACT[a]

No.	Compound	Mol wt
1	4,7-Dimethylbenzofuran	146
2	Benzoic acid	122
3	Nitrogen-containing PAH	177
4	Naphthaldehyde	156
5	1,2-Diphenylethane	182
6	Biphenylamine isomer	169
7	Diphenylmethanone	182
8	1,3-Diphenylpropaneisomer	196
9	Nitrogen-containing PAH	181
10	Biphenylamine isomer	169
11	Unidentified	208
12	9-Fluorenone	180
13	Bis(methylphenyl)diazene	210
14	Phenylbenzoate	198
15	1,3-Diphenyl-2-propen-1-one	208
16	Unidentified	210
17	*o*-Terphenyl	230
18	Chloro-9-fluorenone isomer	214
19	Chloro-9-fluorenone isomer	214
20	Chloro-9-fluorenone isomer	214
21	4H-Cyclopenta[def]phenanthrene-4-one	204
22	Nitrogen-containing PAH	239
23	Dichloro-9-fluorenone isomer	248
24	Dichloro-9-fluorenone isomer	248
25	1-Chloro-9,10-anthraquinone	242
26	*m*-Terphenyl	230
27	*p*-Terphenyl	230
28	Dichloro-9-fluorenone isomer	248
29	Unidentified	244
30	Trichloro-9-fluorenone isomer	282
31	Trichloro-9-fluorenone isomer	282
32	Trichloro-9-fluorenone isomer	282
33	Unidentified	244
34	Unidentified	314
35	7*H*-Benz[de]anthracene-7-one (or 11-benzo[a]fluorenone)	230
36	Methylphenylindole	207
37	Trichloro-9-fluorenone isomer	204
38	Trichloro-9-fluorenone isomer	284
39	Methylphenylindole	207
40	Triphenylene	228

(continued)

TABLE 3 (continued)

No.	Compound	Mol wt
41	Chrysene	228
42	Tetrachloro-9-fluorenone isomer	316
43	Diisooctyl phthalate	390
44	Methylphenyl-1*H*-indole	207
45	Tetrachloro-9-fluorenone isomer	316
46	Benzo[cd]pyrenone	254
47	Pentachloro-9-florenone isomer	350
48	*o*-Quaterphenyl	306
49	Benzo[e]pyrene	252
50	Pentachloro-9-fluorenone isomer	350
51	1,7-Diphenylnaphthalene	280
52	Benzo[a]pyrene	252
53	*m*-Quaterphenyl	306
54	Unidentified	354
55	Unidentified	320

[a] J. Chromatogr. 285: 423-441; 1984.

TABLE 4. COMPOUNDS IDENTIFIED IN HPLC FRACTIONS 4 and 5 OF ONTARIO FLY-ASH EXTRACT[a]

No.	Fraction	Compound	Mol wt
1	4	1-(Methylphenyl)ethanone	134
2		Biphenylamine isomer	169
3		Unidentified	186
4		Unidentified	196
5		Xanthen-9-one	196
6		Ketone derivative of PAH	196
7		Dibutyl phthalate	278
8		9,10-Anthraquinone	208
9		Unidentified	258
10		Quinone derivative of PAH	220
11		Ketone derivative of PAH	230
12		Diisooctyl phthalate	390
13		Nitrogen-containing PAH	209
14		Unidentified phthalate	
1	5	Unidentified	198
2		Diethyl phthalate	222
3		Biphenylamine isomer	169
4		Caffeine	194
5		Dibutyl phthalate	278
6		Benzo[c]cinnoline	134
7		Butylbenzyl phthalate (impurity)	312
8		Unidentified	171
9		Dioctyl phthalate (impurity)	330
10		Unidentified	330

[a] J. Chromatogr. 285: 423-441; 1984.

TABLE 5. CONCENTRATION OF PCDDs DETERMINED IN FLY ASH[a]

Fly ash	Compound	Concentration, ng/g fly ash	Relative standard deviation,[b] %
Ontario	TCDD	541	19
	P_5CDD	467	16
	P_6CDD	591	22
	P_7CDD	434	23
	OCDD	467	19
Oslo	TCDD	Not detected	
	P_5CDD	11	15
	P_6CDD	51	5
	P_7CDD	119	15
	OCDD	186	9

[a] J. Chromatogr. 285: 423-441; 1984.

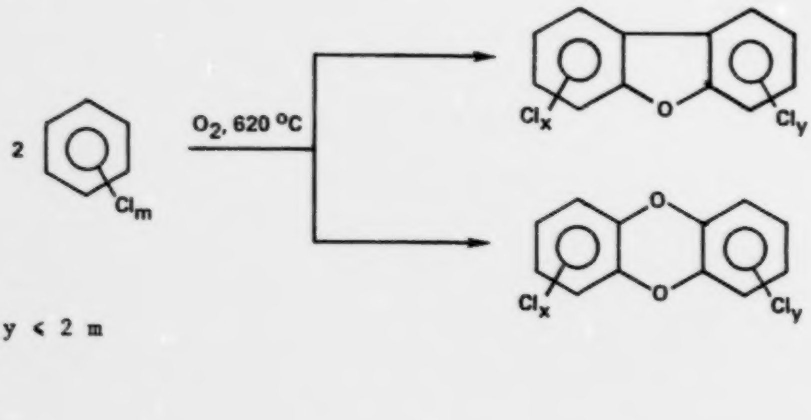
[b] Two determinations.

Lusterhouwer et al. (20) and Cavallaro et al. (21) have suggested that PCDDs and PCDFs may be formed during combustion from chlorinated precursors such as PCBs, PCNs, CBs and polyvinyl chloride. Also these same authors indicate that PCDDs and PCDFs may form in a complex array of reactions between nonchlorinated precursors and inorganic forms of chlorine. Chlorine as HCl and ohter species does occur at low levels in most fuels and materials used for combustion (5). The presence of PCDD in effluents from incinerator processes without sources of chlorinated compounds might be indicative of this process.

Wong (22) traced several trends from the data resulting from the analysis of incinerator effluents. PCDDs, PCDFs, and their precursors were present in municipal refuse and incinerator effluents in trace quantities. After the refuse-burning process, PCDDs increased by 28%, PCDFs increased by a factor of 25, CBs increased by a factor of 31, CPs remained practically unchanged, and PCBs decreased by 94%. From the effluents of municipal incinerator plants analyzed in a study by Ozvacic et al. (23), the magnitude of total output of PCBs, CBs, and CPs varied inversely with the magnitude of total output of PCDDs and PCDFs. The authors concluded that chlorinated compounds were at least partially responsible for the formation of PCDDs and PCDFs. This possibility appears to be the most plausible based on experimental evidence.

Most of the information obtained on the formation of PCDD and PCDF from chlorinated precursors during combustion comes from pyrolysis experiments. Buser, in 1979, reported on the formation of PCDFs and PCDDs through pyrolytic reactions from CBs (24). Tri-, tetra-, and pentachlorobenzene in sealed quartz mini-ampoules were pyrolyzed in the presence of air at 620 °C. Products formed were tetra- to octa- chlorinated dibenzofurans, tetra- to octa- chlorinated dibenzodioxins, chlorophenols, and a series of other chlorinated compounds. CBs were still the major components in pyrolyzed samples. Decomposition was higher for the lower chlorinated species. Greater than 95% of the trichlorobenzenes, ≈90% of the tetrachlorobenzenes, and ≈50% of the pentachlorobenzenes were destroyed. CBs with a higher degree of chlorination than originally present were found in all pyrolysates. For example, tetra- and pentachlorobenzenes were formed from trichlorobenzenes; penta- and hexachlorobenzenes from tetrachlorobenzenes; and hexachlorobenzene from pentachlorobenzene.

Significant amounts of PCDFs and PCDDs are formed via a bimolecular reaction (24):



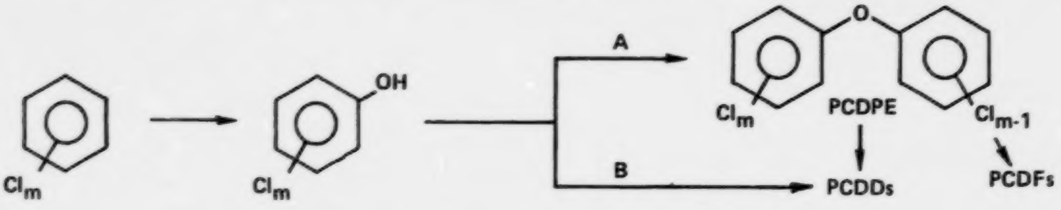
(1)

where

$x + y < 2m$

In general, PCDDs and PCDFs have chlorine numbers of 2m-2, 2m-1, and 2m, where m is the chlorine number of the CB used. Some of the higher CBs produced during pyrolysis yielded some higher chlorinated dibenzofurans and dioxans. The amounts of PCDDs formed were smaller than the amounts of PCDFs formed. For example, from a 200-μg sample of trichlorobenzenes, 400 ng of TCDF was formed as compared with 30 ng of TCDD.

CPs were also products of the pyrolyses, with the degree of chlorination the same or higher than the chlorination of CBs. Scheme 1 illustrates that CPs could be intermediate in the formation of PCDFs and PCDDs:



SCHEME I

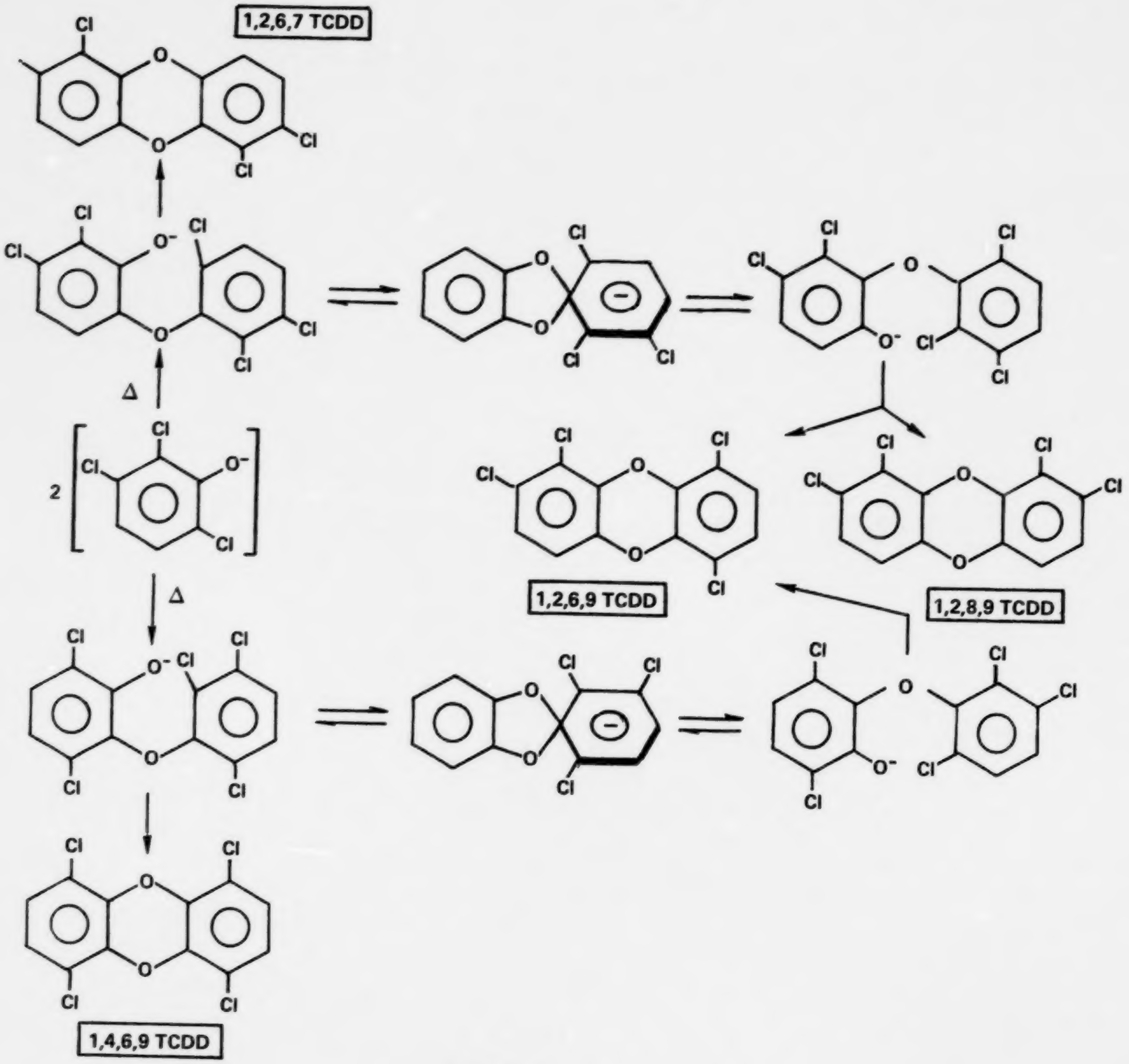
where

PCDPE = polychlorodiphenyl ether

PCDPEs were not observed in the pyrolysis products here; however, dimerization of CPs could occur as a route to PCDPEs. Additional chlorinated compounds found in the pyrolysates were PCNs, polychlorostyrenes, some small quantities of PCBs and compounds tentatively identified as chlorinated benzofurans and dibenzonitriles.

Polychlorinated phenols were mentioned previously as being a precursor to the formation of PCDDs. Approximtely 150,000 tons of CPs are produced annually for use as wood preservations (fungicides), as bactericides in cutting fluids and industrial oils, as herbicides, and as starting materials for products such as the chlorinated phenoxy acids 2,4-D and 3,4,5-T (25). CPs contain a variety of contaminants and by-products. Besides other CPs, polychlorinated phenoxyphenols are present at the percent level. PCDDs, PCDFs, and PCDPEs are present at the 10 to 100-ppm level.

Formation of PCDDs from chlorophenates during pyrolysis has been reported (24-26). Nestrick et al. prepared PCDD standards by the pyrolysis of polychlorinated phenates (27). All 22 of the TCDD isomers were prepared by the pyrolysis of potassium chlorophenates at 280 °C on a silica support matrix. Scheme 2 presents the reaction pathway for the condensation of 2,3,6-trichlorophenol:



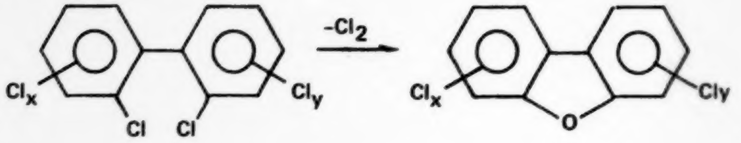
SCHEME 2

Four possible isomers of TCDD can be formed in this pyrolysis of 2,3,6-TCP.

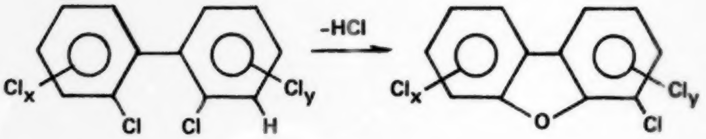
Bridle et al. (30) estimated that significant levels of dioxins and furans could be formed from the combustion of CP-contaminated wood wastes. The penta, hexa and hepta homologues predominated. Thermal regions up to 900 °C and residence times of 3 s were not adequate to destroy these combustion by-products.

PCBs have been reported as being precursors to the formation of PCDFs. Buser (31,32) reports on the formation of PCDFs from pyrolysis of PCB isomers. Reaction mechanisms for the thermal conversion of PCBs to PCDFs have been outlined (25). Intramolecular cyclizations are described, with three reaction routes leading to different PCDF isomers from the same PCB isomer.

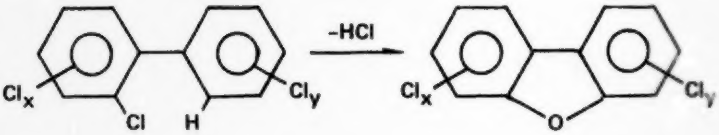
ROUTE 1
(LOSS OF ORTHO-Cl_2)


$-Cl_2$
Cl_x
Cl_y
Cl
Cl
Cl_x
Cly
O

ROUTE 2:
(LOSS OF HCl involving a 2,3-chlorine shift)

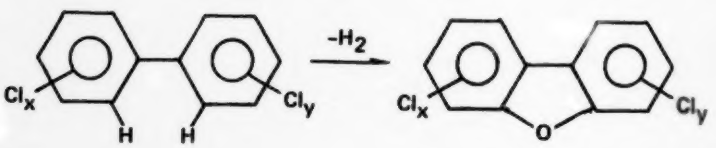

-HCl
Cl_x
Cl_y
Cl
Cl
H
Cl_x
Cl_y
O
Cl

ROUTE 3:
(LOSS OF ORTHO-HCl)

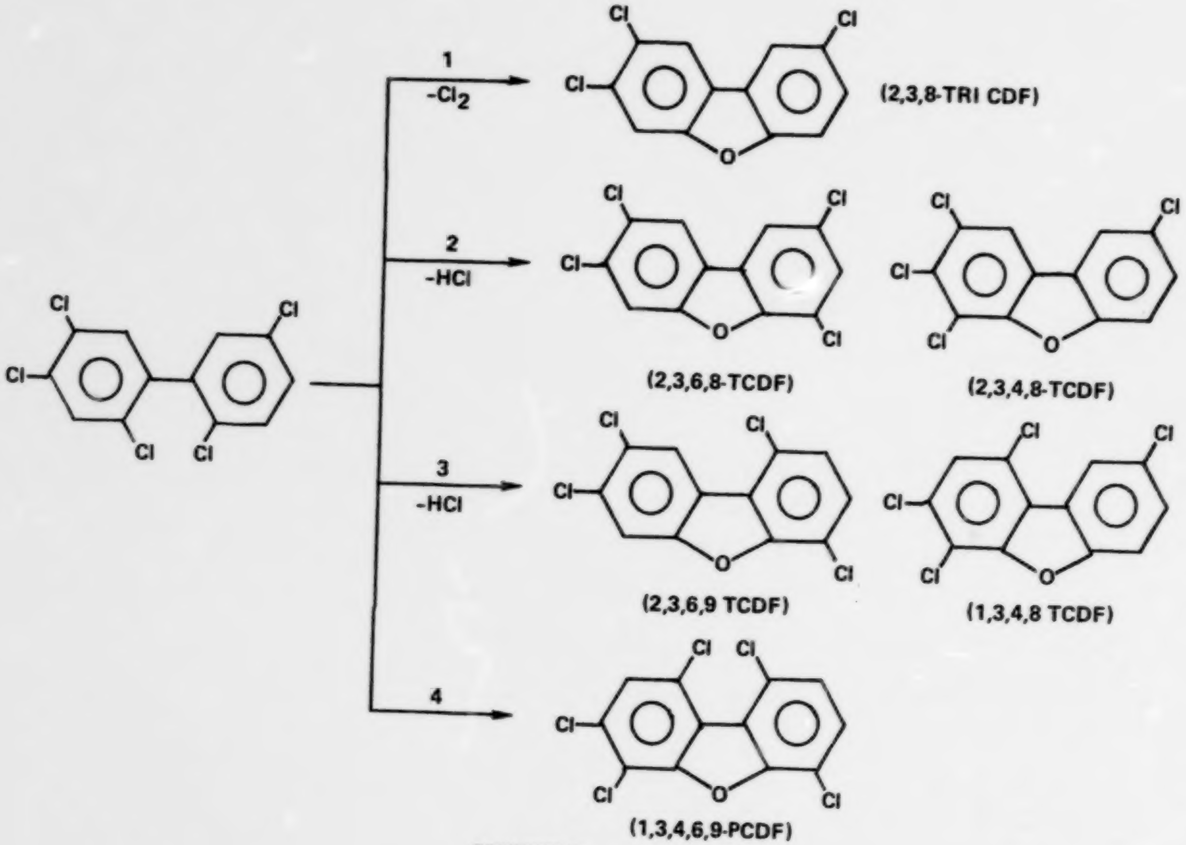

-HCl
Cl_x
Cl_y
Cl
H
Cl_x
Cl_y
O

A fourth route is found to occur with some PCB isomers.

ROUTE 4:
(LOSS OF ORTHO-H_2)


$-H_2$
Cl_x
Cl_y
H
H
Cl_x
Cl_y
O

These reaction paths could be used to predict the formation of specific PCDFs. For example, the pyrolysis of 2,4,5,2',5'-pentachlorobiphenyl shows that all four reaction routes are possible.



SCHEME 3

The PCDFs identified chromatographically by comparison with reference compounds were 2,3,8-tri-CDF and 2,3,6,8-tetra-CDF. No attempts were made to identify the remaining tetra-CDFs. A small amount of penta-CDF was observed (25).

Buser (31) contends that the PCDFs found in fly ash and flue gases from municipal incinerators and industrial heating facilities originated from PCBs present in the wastes. Most of the major PCDFs found in the fly-ash samples analyzed by Buser (31) were also formed in the pyrolysis of commercial PCBs, Aroclor 1254 and 1260. PCNs were also found in smaller amounts, along with other chlorinated compounds.

Tiernan et al.(33) completed a risk assessment on two incinerator facilities employed for PCB destruction tests. They found that, in general, the quantities of PCDFs exceeded the quantities of corresponding PCDDs. They concluded that the risks were negligible (risks of additional cancers in exposed populations ranged from 1 to 4 in 10,000,000 in one facility and 1 to 180 in 10,000,000 in the other). Under proper conditions, incineration should be a viable procedure for destruction of PCB wastes. These conditions included a residence time in the primary incineration chamber of 2.2 s with a temperature of 1232 to 1282 °C and a residence time of 2 s in the secondary chamber with a temperature of 760 °C.

Laboratory pyrolysis experiments dealing with the formation of PCDDs and PCDFs from CPs, chlorophenates, CBs, and PCBs, cannot be considered adequate to demonstrate that PICs would form in gas-phase processes. Shaub and Tsang (34) suggested that these formations may be due in part to surface reactions.

Shaub and Tsang (34) also proposed a model mechanism for the homogeneous gas-phase formation of PCDDs from CPs in an incinerator environment. By drawing on existing information about gas-phase reaction processes, numerical calculations were made. The results indicated that gas-phase formation of PCDDs had a low probability at high temperatures (>1200 K) when mixing between fuel and air was sufficient. However, in actual practice, PCDD emissions sometimes exceeded the levels predicted by the above results.

There were several possible reasons given for this discrepancy (34). PCDDs are originally present in the input waste stream and survive thermal destruction in significant amounts. However, gas-phase thermal-destruction efficiency for PCDDs is extremely high at flame temperatures typically achieved in an incinerator, $T > 1500$ K. Because of mixing inhomogeneities, some unburned chloroorganics survive the main combustion environment and make it into the post-combustion zone. Temperature fluctuations (thermal transients) in the incinerator result in incomplete thermal stressing because of gas entrapment in cold pockets, promoting PCDD production at above ambient but below flame-temperature conditions. Well-designed incinerators would reduce the possibility of gas entrapment. Molecules other than CPs may be converted to PCDD in the gas phase. These molecules would, however, be amenable to the same type of numerical analysis presented for the CPs.

PCDDs could be formed in the gas phase by chlorination of organics by inorganic chlorine sources in municipal-waste-input feed streams. The presence of $FeCl_2$, $FeCl_3$, and NaCl in the gas phase has been investigated. Very high temperatures, in excess of 1000 K, were found to be required for even low levels of NaCl to build up in the gas phase, and temperatures in excess of 1200 K were required for low levels of iron chlorides to develop in the gas phase. Heavy-metal chlorides such as $ZnCl_2$, $CdCl_2$, and $PbCl_2$ are much more volatile at these temperatures, but except for fairly specific waste streams the amounts of these metals initially present is low. Even at these high temperatures, PCDD formation would involve multistep processes with several reaction steps. The reactant concentrations are low and cannot easily compete with other routes that would lead to destruction during the combustion process. Synthesis from inorganic precursors would, therefore, seem to be unimportant in the gas phase.

Non-gas-phase reactions or a combination of gas-phase and non-gas-phase reactions may contribute to production of PCDD during the combustion process (34). This possibility is intriguing. It indicates that reactions involving suspended fly ash or reactive processes within or on the grate beds of incinerators may have to be considered. Fly ash is a mixture of silica and metal oxides and should have many reactive Lewis and Brønsted acid-base sites which could promote reactions at temperatures considerably below those at which destruction processes dominate.

The formation of PICs in an incinerator environment is probably a very complex phenomena, impacted by several factors. The two classes of PICs which are under close scrutiny are PCDFs and PCDDs. PCDDs and PCDFs are apparently formed from precursors in the hazardous-waste load such as PCBs, PCNs, CBs, CDs, polyvinyl chloride, and other chlorinated organic compounds. However, some of these compounds may also be formed from other chlorinated organics during the incineration process. Incinerator conditions also play a major role in the extent of PIC formation.

Analytical methodology will need to be reviewed for the PICs listed in this report as well as other PICs. Interferences in the determination of PICs in fly ash as well as in the waste feed will also need to be carefully assessed. It would also be of interest to understand the nature of the surface reactions and reactions in solids (such as fly ash) which appear to be contributing to the formation of PICs in the incineration process.

Defining and maintaining proper operating conditions for the incinerator should reduce the formation of PICs in the incinerator effluent.

SECTION 5

TECHNICAL APPROACH TO APPLYING THE GENERALIZED ANALYSIS METHODS TO PICs AND POHCs

GENERAL CONSIDERATIONS FOR THE DETERMINATION OF PICs

The primary goal of our experimental work has been to develop generalized analytical methods for the determination of PICs in appropriate organic solvents. Capillary gas chromatography was chosen as the basic analysis technique. During the development of the capillary GC methods, both flame-ionization detection (FID) and mass spectrometry (MS) were used as detection systems.

We have concentrated on applying previously developed POHC general analysis methods to the analysis of PICs. A large body of the literature on PICs is based on the findings of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) in incinerator effluents. We have evaluated generalized GC/FID and GC/MS analysis methods for PCDDs and PCDFs. The methods are ones we previously developed, and will be used to analyze for specific POHCs as well as for screening studies. We wished to define how well the methods would work for dioxins and furans if one simply "looked for them" during an analysis that might be primarily for another compound. If one were to choose a method for only dioxins or furans, then one of the methods previously reported by Harless, Tiernan or others would probably be the method of choice (especially if the detection limit needed to be pushed as low as possible).

The work described in this section reports on 36 compounds selected as candidate PICs by GC/FID and GC/MS. A detailed account of our findings is given in Section 6 of this report.

SELECTION OF CANDIDATE PICs

We selected candidate PICs for analysis by GC/FID and GC/MS from the literature review given in Section 4 of this report and from discussions with EPA. We chose 10 polychlorinated dibenzo-p-dioxins and 3 polychlorinated dibenzofurans from commercially available sources. We also chose 23 substituted PAHs from commercially available sources.

PREPARATION OF STOCK STANDARD SOLUTIONS

We prepared stock standard solutions of the candidate compounds in appropriate solvents at a concentration of about 0.05 mg/mL. These stock solutions were serially diluted as required to prepare working standards. Methylene chloride, toluene, and isooctane were used as solvents.

PICs GC ANALYSIS PROCEDURES

Description of Instruments and General Operating Conditions

We developed the GC/MS generalized test method on the Hewlett-Packard Model 5985 gas chromatograph/mass spectrometer-data system. The components of the instruments include a hyperbolic quadrupole mass filter with a convertible electron-impact (EI) and positive-ion chemical-ionization source and a capillary and jet separator GC/MS interface. It also has a data system that includes an HP2113 computer, a high-speed printer, a magnetic tape system, a 50-megabyte HP7920M disk-drive system, a communications interface, GC/MS operating software, and an unabridged NBS spectral library. The supplemental GC/FID work was performed on a Hewlett-Packard Model 5840 gas chromatograph that was equipped for use with capillary columns.

The generalized GC/MS, GC/FID procedure is listed below:

• Instruments:	HP 5985A GC/MS; HP 5840 GC/FID
• Columns:	Two matched SE-54 bonded-fused-silica capillary columns, 0.32-mm ID, 25-m length
• Carrier gas:	Helium, 30 cm/s (2 mL/min)
• Injection:	2 μL splitless
• Column-temperature program:	60 °C for 3 min, then programmed at 20 °C/min to 300 °C, and held at 300 °C for 15 min
• Injection temperature:	290 °C
• FID temperature:	300 °C
• MS parameters:	Scan 41 to 500 amu/s. Electron ionization at 70 eV. Source temperature set to 200 °C. Capillary column plumbed directly into the ion source.

The initial operating conditions chosen were a synthesis of conditions that would allow the separation and detection of as many compounds as possible. Modifications of temperatures and rates may be necessary or desirable to optimize the analysis for specific compounds.

Selected PICs were also analyzed using selected-ion monitoring (SIM) GC/MS to improve the detection limits obtainable. The generalized procedure is listed below:

- Instrument: HP 5985A GC/MS
- Column: Bonded SE-54 fused-silica capillary column, 0.32-mm I.D., 25-m length
- Carrier gas: Helium, 30 cm/s
- Injection: 2 µL splitless
- Column-temperature program: 60 °C for 3 min, then programmed at 20 °C/min to 300 °C, and held at 300 °C for 15 min
- Injection temperature: 290 °C
- MS conditions: Electron ionization at 70 eV. Source temperature set to 200 °C. Capillary column plumbed directly into the ion source. 70 ms/scan dwell time for each mass.

Table 6 summarizes the ions used in the SIM studies.

Optimization of the GC/FID and GC/MS Procedures

GC operating conditions were optimized by analyzing solutions containing selected PICs by the GC/FID technique. The column-head pressure was adjusted appropriately to allow adequate separation of the compounds of interest in less than 30 min.

Having established GC operating conditions by the GC/FID procedure, we then applied the method to the determination of the candidate PICs by GC/MS. The mass spectrometer was operated in a full mass scanning range (41 to 500 amu) in the EI mode. The scan time was maintained at ≈1 s to enable the collection of enough scans to characterize each capillary GC peak. During SIM, the mass spectrometer dwelled on each selected mass for 70 ms/scan.

GC Quality-Control Procedures

We calibrated the GC/FID and the GC/MS procedures with standard solutions of the candidate PICs using anthracene-d_{10} as the internal standard. Five- to eight-point calibration curves were prepared for each candidate PIC determined by gas chromatography. Each curve was a plot of the FID or the MS response (relative to anthracene-d_{10}) as a function of the quantity of the particular PIC injected on the GC column. Appendix A of reference 35 was used as a guideline for estimating detection limits for each candidate POHC. Step 1c, which is quoted below, was selected to develop the estimate of detection limit.

TABLE 6. IONS USED IN SELECTED-ION MONITORING OF SELECTED PICs

Compounds/Classes	Dioxin Ions		Furan Ions		Internal standard $^{13}C_{12}$-TCDD		Internal standard anthracene-d_{10}
Mono-substituted PCDDs and PCDFs	218	220	202	204	332	334	188
Di-substituted PCDDs and PCDFs	252	254	236	238	332	334	188
Tri-substituted PCDDs and PCDFs	286	288	270	272	332	334	188
Tetra-substituted PCDDs and PCDFs	320	322	304	306	332	334	188
Hexa-substituted PCDDs and PCDFs	388	390	372	374	332	334	188
Octa-substituted PCDDs and PCDFs	458	460	442	444	332	334	188

The concentration value that corresponds to the region of the standard curve where there is a significant change in sensitivity at low analyte concentrations, i.e., a break in the slope of the standard curve, can be used as an estimate of the detection limit.

The precision of determinations by GC/FID and GC/MS was estimated by triplicate injections of at least one standard solution of each PIC investigated by gas chromatography. The estimate of precision of the replicate measurements is expressed as the relative standard deviation (RSD) calculated from Equation 2:

$$\mathrm{RSD}\ (\%) = \left(\frac{SD}{\overline{X}}\right) 100 \qquad (2)$$

Standard deviation (SD) was calculated from Equation 3:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (X_1 - \overline{X})^2}{(n - 1)}} \qquad (3)$$

Mean relative response $(\overline{X})$ was calculated from Equation 4:

$$\overline{X} = \frac{1}{n} \sum_{i=1}^{n} X_i \qquad (4)$$

where

n = number of replicate measurements

X_i = relative response of the PIC standard

Furthermore, we used anthracene-d_{10} as an internal standard in the GC methods. Along with the analytes, we injected anthracene-d_{10} to minimize the effect of injection volume and instrument performance upon the precision of determinations. Also, the anthracene-d_{10} internal standard was used to calculate the relative response factor (RRF) for each PIC from Equation 5.

$$\mathrm{RRF} = (A_s C_{is})/(A_{is} C_s) \qquad (5)$$

where

A_s = area of the characteristic ion for the PIC to be measured

A_{is} = area of the characteristic ion for the internal standard

C_{is} = concentration of the internal standard (μg/mL)

C_s = concentration of the PIC (μg/mL) to be measured

Recovery data were not applicable for this phase of the project because our analyses were done with neat solutions and standard mixtures of PICs. We did not collect, prepare, or analyze field samples that required spiking of the matrix with surrogate standards or specific PICs.

GENERAL CONSIDERATIONS FOR THE DETERMINATION OF POHCs

The primary goal of our experimental work has been to develop generalized analytical methods for the determination of POHCs in appropriate organic solvents. Two basic chromatographic techniques were chosen: GC and HPLC. During the development of the capillary GC methods, both FID and MS were used to establish the techniques.

In previous work (Work Assignment 001 and Work Assignment 001, Amendment 1, Contract 68-02-3696, and Work Assignment 111, Contract 68-02-2685), we evaluated GC/MS and HPLC/UV methods for the determination of 70, 38, and 100 POHCs, respectively. The methods were calibrated for candidate organic compounds over concentration ranges of interest and demonstrated acceptable precision in the determination of most of the compounds.

The work described in this report extends the coverage of the previous reports to an additional 59 POHCs by GC. A detailed account of our findings is given in Section 6 of this report.

SELECTION OF CANDIDATE POHCs

We selected candidate POHCs for analysis by GC from the list of compounds in an amendment to Appendix VIII (2). The generalized methods were applied to an additional 59 compounds. Calibration curves, precision data, and detection estimations were obtained for these compounds.

PREPARATION OF STOCK STANDARD POHC SOLUTIONS

We prepared stock standard solutions of the candidate compounds in appropriate solvents at a concentration of either about 2 mg/mL or about 100 mg/mL. These stock solutions were serially diluted as required to prepare working standards. We used methylene chloride, hexane, methanol, or carbon disulfide as the solvent in the preparation of standards for GC/FID and GC/MS studies.

POHCs GC ANALYSIS PROCEDURES

Description of Instruments and General Operating Conditions

We developed the GC/MS generalized test method on the Hewlett-Packard Model 5985 GC/MS-data system and the Hewlett-Packard Model 5840 GC/FID.

The work with both the GC/MS and the GC/FID involved capillary column chromatography with matched, crosslinked fused-silica SE-54 capillary columns 25 m long, 032-mm ID. The initial operating conditions were chosen as a synthesis of the conditions given for several capillary GC methods in the Methods Manual. The initial starting column temperature was 40 °C; the temperature was then programmed at 10 °C to 280 °C for 15 min. Alternate column-temperature programs used rates of 20 °C/min and 30 °C/min. Injection temperature was 290 °C.

The ion source of the mass spectrometer was held at 200 °C and was operated at 70 eV. The FID detector was maintained at 300 °C. The carrier gas was helium and was maintained at a flow rate of ≈2 mL/min. In both the GC/MS work and the GC/FID work, the "splitless" injection technique was employed for all solvents used except methanol. We assumed in the "splitless" injection technique that essentially all of the injected sample reached the column. For compounds dissolved in methanol, a 1:10 split ratio was used.

Optimization of the GC/FID and the GC/MS Procedures

GC operating conditions were optimized by analyzing solutions containing a variety of the candidate POHCs by the GC/FID technique. The column head pressure was adjusted appropriately to allow adequate separation of the compounds of interest in less than 30 min. Having established GC operating conditions by the GC/FID procedure, we then applied the method to the determination of the candidate POHCs by GC/MS. The mass spectrometer was operated in a full mass scanning range in the EI mode. The scan range was from 41 to 450 amu except for hexabromobiphenyl and 1,1a,2,2,3,3a,4,5,5,5a,5b,6-dodecachlorooctahydro-1,3,4-metheno-1*H*-cyclobuta[cd]pentalene. For these compounds the range was 41 to 650 amu. The scan time was maintained at ≈1 s to enable the collection of enough scans to characterize each capillary GC peak.

GC Quality-Control Procedures

The same quality-control procedures were applied to the candidate POHCs as were applied to the candidate PICs.

SECTION 6

RESULTS AND DISCUSSION OF THE DEVELOPMENT OF ANALYSIS METHODS

GC/FID AND GC/MS ANALYSIS OF CANDIDATE PICs

We completed the determination of 36 PICs by GC/FID and GC/MS. In Table 7 we present a summary of the GC/FID determinations of candidate PICs. The retention time and on-column detection limit are given for each compound. The compounds are listed in the order of their elution from the GC column. Retention times are relative to that observed for the internal standard, anthracene-d_{10}. The on-column detection limit was estimated using experimentally determined calibration curves as suggested in Appendix A of Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA 600/4-82-057, July 1982 (35). Typical values were 2 to 40 ng. Reference chromatograms and calibration curves, which appear in Appendix A of this report, are also cited in Table 7.

Appendix A contains chromatograms of individual PICs and PIC mixtures. The chromatograms demonstrate the observed retention times, the peak shapes, and any shifts in the base line that occurred during the analysis. Although no two candidate PICs may coexist in a field situation and, therefore, may not require simultaneous determination, the chromatogram of the compound mixture demonstrates the resolving power of the capillary column in general.

Each reference calibration curve given in Appendix A and cited in Table 1 is a point-to-point plot (solid line) of the FID response ratio as a function of the quantity of the analyte injected onto the GC column. Each plot can be used to estimate the sensitivity expected in the determination of a particular analyte. In generating these curves, we made no attempt to determine the upper limit of the linear range of determinations. However, each plot demonstrates the linearity of the FID response within the range of quantities investigated. A laboratory that uses these methods will have to generate its own calibration curves, which may differ slightly from the ones given in this report. Our calibration curves will serve as a guide.

Also, with each calibration curve is the least-squares plot (a broken line) based on the linear-regression analysis of FID response versus concentration of analyte.

In Table 8, we summarize the GC/MS determination of candidate PICs. As in Table 7, the rentention times are given relative to the internal standard, anthracene-d_{10}. Table 8 also lists the five most abundant mass fragments of each compound and, moreover, specifies the mass of the ion of each compound that was used for the establishment of detection limits and for the generation of calibration curves. The detection limits were estimated from the calibration curves. Typical values of the detection limits were 3.0 to 50 ng.

TABLE 7. SUMMARY OF GC/FID DETERMINATIONS OF CANDIDATE PICs

Compound	Relative retention time[a]	On-column detection limit, ng	Appendix A Reference chromato-gram	Appendix A Reference calibration curve
1-Fluoronaphthalene	0.619	4	A-22	A-23
2-Bromonaphthalene	0.820	5	A-1	A-2
3-Bromoquinoline	0.825	2	A-22	A-24
1-Bromo-2-methylnaphthalene	0.896	3	A-1	A-3
1-Iodonaphthalene	0.899	2	A-34	A-35
1-Bromo-4-methylnaphthalene	0.907	3	A-22	A-25
2-(Bromomethyl)naphthalene	0.929	9	A-1	A-4
1-Amino-4-chloronaphthalene	0.972	43	A-34	A-36
2-Chlorodibenzo-*p*-dioxin	0.980	10	A-17	A-18
1-Chlorodibenzo-*p*-dioxin	0.983	2	A-9	A-10
9-Bromofluorene	0.991	10	A-34	A-37
5-Bromoacenaphthene	1.01	5	A-22	A-26
1-Amino-4-bromonaphthalene	1.02	4	A-34	A-38
1-Chloro-8-nitronaphthalene	1.03	4	A-22	A-27
2-Chloroanthracene	1.05	7	A-34	A-39
3,6-Dichlorodibenzofuran	1.06	3	A-20	A-21
2-Bromofluorene	1.06	10	A-22	A-28
2,7-Dichlorodibenzo-*p*-dioxin	1.08	2	A-9	A-11
2,3-Dichlorodibenzo-*p*-dioxin	1.08	10	A-17	A-19
1-Chloroanthracene	1.11	4	A-1	A-5
9-Chloroanthracene	1.11	4	A-22	A-29
9-(Chloromethyl)anthracene	1.11	4	A-34	A-40
1,2,4-Trichlorodibenzo-*p*-dioxin	1.16	5	A-9	A-12
9-Bromophenanthrene	1.16	4	A-1	A-6
9-Bromoanthracene	1.17	5	A-22	A-30
9,10-Dichloroanthracene	1.20	4	A-1	A-7
2,3,7,8-Tetrachlorodibenzofuran	1.23	2	A-44	A-45
1,2,3,4-Tetrachlorodibenzo-*p*-dioxin	1.28	2	A-9	A-13
6,9-Dichloro-2-methoxyacridine	1.28	5	A-1	A-8
9,10-Dibromoanthracene	1.30	9	A-34	A-41
3,6-Dibromocarbazole	1.37	8	A-22	A-31
1,2,3,4,7-Pentachlorodibenzo-*p*-dioxin	1.40	10	A-42	A-43
1,2,3,4,7,8-Hexachlorodibenzo-*p*-dioxin	1.57	8	A-9	A-14
1,2,3,4,6,7,8-Heptachlorodibenzo-*p*-dioxin	1.83	40	A-32	A-33
1,2,3,4,6,7,8,9-Octachlorodibenzo-*p*-dioxin	2.15	20	A-9	A-15
1,2,3,4,6,7,8,9-Octachlorodibenzofuran	2.17	20	A-9	A-16

[a] Relative to the retention time of anthracene-d_{10}.

Reference total-ion chromatograms, calibration curves, and mass spectra are cited in Table 8. The mass spectra are presented in Appendix B along with the reference chromatograms and calibration curves.

In Table 9, we present the results of triplicate GC/FID determinations of all of the candidate PICs listed in Table 7. The calculated values of the standard deviation (SD) and relative standard deviation (RSD) in Table 9 indicate that most GC/FID determinations gave acceptable precision. The RSDs ranged from 0.2% to 9%.

In Table 10, we present the results of triplicate GC/MS determinations of all of the PICs listed in Table 8. In general, the precision of the GC/MS determinations was poorer than the GC/FID determinations. The RSDs ranged from 3% to 27%.

In Table 11, we present the estimated levels of detection between the standard mass acquire program and the selected-ion monitoring conditions for selected PCDDs and OCDF. The estimated limits of detection were 40 to 200 times lower using selected-ion monitoring.

GC/FID AND GC/MS ANALYSIS OF CANDIDATE POHCs

We completed the determination of 56 POHCs by GC/FID and 58 POHCs by GC/MS. In Table 12, we present a summary of the GC/FID determinations of candidate POHCs. The retention time and on-column detection limits are given for each compound. The compounds are listed in the order of their elution from the GC column. Retention times are relative to that observed for the internal standard, anthracene-d_{10}. The on-column detection limit was estimated using the calibration curve as suggested in Appendix A of Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA 600/4-82-057, July 1982. Typical values were 20 to 800 ng. Reference chromatograms and calibration curves, which appear in Appendix C, are also cited in Table 12.

Appendix C contains chromatograms of individual POHCs and POHC mixtures. The chromatograms demonstrate the observed retention times, the peak shapes, and any shifts in the base line that occurred during the analysis. Although no two of the candidate PICs may coexist in a field situation and, therefore, may not require simultaneous determination, the chromatogram of the compound mixtures demonstrates the resolving power of the capillary column in general.

Each reference calibration curve given in Appendix C and cited in Table 12 is a point-to-point plot (solid line) of the FID response ratio as a function of the quantity of the analyte injected onto the GC column. Each plot can be used to estimate the sensitivity expected in the determination of a particular analyte. In generating these curves, we made no attempt to determine the upper limit of the linear range of determinations. However, each plot demonstrates the linearity of the FID response within the range of quantities investigated. A laboratory that uses these methods will have to generate its own calibration curves, which may differ slightly from the ones given in this report. Our calibration curves will serve as a guide.

TABLE 8. SUMMARY OF GC/MS DETERMINATIONS OF CANDIDATE PICs

Compound	Relative retention time[a]	On-column detection limit, ng	Mol wt	Masses of characteristic EI ions (relative abundance)					Appendix B		
				1	2	3	4	5	Reference chromatogram	Reference mass spectrum	Reference calibration curve
1-Fluoronaphthalene	0.565	9	146	146(100)	73(10)	147(10)	145(8)	125(8)	B-35	B-36	B-37
2-Bromonaphthalene	0.792	5	206	208(100)	206(90)	127(55)	126(14)	207(12)	B-1	B-2	B-3
3-Bromoquinoline	0.801	4	207	207(100)	209(94)	128(89)	101(33)	75(22)	B-35	B-38	B-39
1-Bromo-2-methylnaphthalene	0.880	50	220	141(100)	220(89)	222(86)	139(34)	115(32)	B-1	B-4	5
1-Iodonaphthalene	0.882	5	254	127(100)	254(98)	126(19)	255(12)	128(10)	B-57	B-58	B-59
1-Bromo-4-methylnaphthalene	0.893	5	220	141(100)	220(97)	222(93)	139(34)	115(26)	B-35	B-40	B-41
2-(Bromomethyl)naphthalene	0.920	4	220	141(100)	115(17)	139(14)	70(13)	142(12)	B-1	B-6	B-7
1-Chlorodibenzo-p-dioxin	0.981	9	218	218(100)	220(34)	219(14)	155(12)	221(5)	B-16	B-17	B-18
2-Chlorodibenzo-p-dioxin	0.984	10	218	218(100)	220(34)	155(30)	127(23)	109(18)	B-27	B-28	B-29
1-Amino-4-chloronaphthalene	0.994	9	177	177(100)	179(32)	115(28)	178(13)	149(11)	B-57	B-60	B-61
5-Bromoacenaphthene	1.02	10	232	153(100)	152(83)	76(67)	232(39)	234(38)	B-35	B-42	B-43
9-Bromofluorene	1.03	10	244	165(100)	166(15)	163(14)	82(13)	164(10)	B-57	B-62	B-63
1-Chloro-8-nitronaphthalene	1.04	9	207	172(100)	126(72)	207(40)	161(37)	149(25)	B-35	B-44	B-45
1-Amino-4-bromonaphthalene	1.06	43	221	223(100)	221(94)	115(51)	70(24)	114(16)	B-57	B-64	B-65
3,6-Dichlorodibenzofuran	1.07	3	236	236(100)	238(70)	173(20)	237(15)	240(12)	B-32	B-33	B-34
2-Bromofluorene	1.07	5	244	165(100)	246(63)	244(59)	163(24)	166(18)	B-35	B-46	B-47
2,7-Dichlorodibenzo-p-dioxin	1.10	9	252	252(100)	254(66)	189(28)	126(25)	253(13)	B-16	B-19	B-20
2,3-Dichlorodibenzo-p-dioxin	1.10	3	252	252(100)	254(63)	126(22)	189(22)	253(14)	B-27	B-30	B-31
1-Chloroanthracene	1.12	4	212	212(100)	214(32)	176(26)	88(20)	213(15)	B-1	B-8	B-9
2-Chloroanthracene	1.12	7	212	212(100)	214(37)	176(29)	177(18)	213(17)	B-57	B-66	B-67
9-Chloroanthracene	1.13	8	212	212(100)	214(33)	176(30)	88(18)	213(15)	B-35	B-48	B-49
9-Bromophenanthrene	1.18	8	256	256(100)	258(99)	176(57)	88(48)	177(34)	B-1	B-10	B-11
9-Bromoanthracene	1.19	10	256	256(100)	258(98)	88(61)	176(54)	177(29)	B-35	B-50	B-51
1,2,4-Trichlorodibenzo-p-dioxin	1.20	10	286	288(100)	286(89)	290(34)	223(23)	225(20)	B-16	B-21	B-22
9-(Chloromethyl)anthracene	1.22	7	226	191(100)	189(28)	226(21)	192(18)	94(17)	B-57	B-68	B-69
9,10-Dichloroanthracene	1.23	4	246	246(100)	248(73)	176(33)	247(18)	87(15)	B-1	B-12	B-13
2,3,7,8-Tetrachlorodibenzofuran	1.24	9	304	306(100)	304(77)	308(45)	307(11)	310(10)	B-75	B-76	B-77
1,2,3,4-Tetrachlorodibenzo-p-dioxin	1.31	10	320	322(100)	320(78)	324(49)	323(12)	257(11)	B-16	B-23	B-24

(continued)

TABLE 8. SUMMARY OF GC/MS DETERMINATIONS OF CANDIDATE PICs

Compound	Relative retention time[a]	On-column detection limit, ng	Mol wt	Masses of characteristic EI ions (relative abundance)					Appendix B		
				1	2	3	4	5	Reference chromatogram	Reference mass spectrum	Reference calibration curve
6,9-Dichloro-2-methoxyacridine	1.340	10	277	277(100)	234(78)	279(69)	236(51)	164(23)	B-1	B-14	B-15
9,10-Dibromoanthracene	1.360	4	334	176(100)	88(84)	336(52)	87(37)	334(26)	B-57	B-70	B-71
1,2,3,4,7-Pentachlorodibenzo-*p*-dioxin	1.390	10	354	356(100)	354(71)	358(63)	293(26)	291(22)	B-72	B-73	B-74
3,6-Dibromocarbazole	1.420	4	323	325(100)	323(54)	327(52)	246(24)	244(24)	B-35	B-52	B-53
1,2,3,4,7,8-Hexachlorodibenzo-*p*-dioxin	1.490	8	388	390(100)	392(86)	388(50)	394(36)	327(30)	B-16	B-25	B-26
1,2,3,4,6,7,8-Heptachlorodibenzo-*p*-dioxin	1.590	10	422	424(100)	426(99)	428(50)	422(41)	361(28)	B-54	B-55	B-56
1,2,3,4,6,7,8,9-Octachlorodibenzo-*p*-dioxin	1.680	40	456	460(100)	458(96)	142(77)	462(74)	167(57)	B-75	B-78	B-79
1,2,3,4,6,7,8,9-Octachlorodibenzofuran	1.690	30	440	442(100)	444(98)	446(72)	440(41)	154(40)	B-75	B-80	B-81

[a] Relative to the retention time of anthracene-d_{10}.

TABLE 9. PRECISION OF GC/FID DETERMINATIONS

Compound	Quantity injected, ng	RRF[a]		
		Mean	SD	RSD, %
1-Amino-4-bromonaphthalene	42.0	0.548	0.0055	0.99
1-Amino-4-chloronaphthalene	42.6	0.0281	0.00012	0.42
5-Bromoacenaphthene	51.0	0.554	0.0011	0.20
9-Bromoanthracene	50.8	0.529	0.0053	1.0
2-Bromofluorene	48.8	0.595	0.0039	0.65
9-Bromofluorene	49.6	0.379	0.0050	1.3
1-Bromo-2-methylnaphthalene	58.8	0.635	0.0050	0.79
1-Bromo-4-methylnaphthalene	54.2	0.664	0.0070	1.1
2-(Bromomethyl)naphthalene	44.2	0.431	0.0095	2.2
2-Bromonaphthalene	46.0	0.733	0.0080	1.1
9-Bromophenanthrene	41.2	0.528	0.011	2.0
3-Bromoquinoline	41.0	0.543	0.0071	1.3
1-Chloroanthracene	40.0	0.743	0.017	2.3
2-Chloroanthracene	33.8	0.356	0.0015	0.42
9-Chloroanthracene	38.4	0.678	0.0083	1.2
1-Chlorodibenzo-p-dioxin	17.8	0.474	0.0013	0.28
2-Chlorodibenzo-p-dioxin	31.1	0.659	0.0014	0.22
9-(Chloromethyl)anthracene	36.4	0.945	0.0034	0.36
1-Chloro-8-nitronaphthalene	43.6	0.560	0.0058	1.0
9,10-Dibromoanthracene	42.6	0.318	0.0090	2.8
3,6-Dibromocarbazole	41.0	0.178	0.017	9.3
9,10-Dichloroanthracene	42.0	0.638	0.017	2.6
3,6-Dichlorodibenzofuran	30.4	0.626	0.0044	0.71
2,3-Dichlorodibenzo-p-dioxin	31.2	0.553	0.0023	0.42
2,7-Dichlorodibenzo-p-dioxin	17.1	0.388	0.0020	0.52
6,9-Dichloro-2-methoxyacridine	51.0	0.318	0.012	3.8
1-Fluoronaphthalene	42.8	0.928	0.012	1.3
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	80.4	0.118	0.0023	2.0
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	15.1	0.190	0.011	6.0
1-Iodonaphthalene	45.6	0.540	0.0022	0.42
1,2,3,4,6,7,8,9-Octachlorodibenzofuran	17.1	0.104	0.0073	7.0
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin	17.3	0.0901	0.0077	8.5
1,2,3,4,7-Pentachlorodibenzo-p-dioxin	32.8	0.379	0.0094	2.5
2,3,7,8-Tetrachlorodibenzofuran	22.5	0.356	0.0050	1.4
1,2,3,4-Tetrachlorodibenzo-p-dioxin	19.4	0.277	0.0077	2.8
1,2,4-Trichlorodibenzo-p-dioxin	16.8	0.352	0.0043	1.2

[a] RRF = $(A_sC_{is}/A_{is}C_s)$.

TABLE 10. PRECISION OF GC/MS DETERMINATIONS

Compound	Quantity injected, ng	RRF[a]		
		Mean	SD	RSD, %
1-Amino-4-bromonaphthalene	42.8	0.116	0.024	21
1-Amino-4-chloronaphthalene	42.6	0.245	0.037	15
5-Bromoacenaphthene	51.0	0.328	0.068	21
9-Bromoanthracene	50.8	0.583	0.046	7.8
2-Bromofluorene	48.8	0.606	0.092	15
9-Bromofluorene	49.6	0.452	0.035	7.6
1-Bromo-2-methylnaphthalene	58.8	0.206	0.016	7.6
1-Bromo-4-methylnaphthalene	54.2	0.246	0.026	11
2-(Bromomethyl)naphthalene	44.2	0.479	0.0049	1.0
2-Bromonaphthalene	46.0	0.363	0.025	7.0
9-Bromophenanthrene	41.2	0.520	0.040	7.8
3-Bromoquinoline	41.0	0.300	0.33	11
1-Chloroanthracene	40.0	0.987	0.033	3.4
2-Chloroanthracene	33.8	1.35	0.12	8.7
9-Chloroanthracene	38.4	0.923	0.086	9.3
1-Chlorodibenzo-*p*-dioxin	17.8	0.297	0.018	6.2
2-Chlorodibenzo-*p*-dioxin	31.1	0.827	0.076	9.2
9-(Chloromethyl)anthracene	36.4	0.148	0.019	13
1-Chloro-8-nitronaphthalene	43.6	0.342	0.043	13
9,10-Dibromoanthracene	42.6	0.398	0.089	22
3,6-Dibromocarbazole	41.0	0.235	0.010	4.3
9,10-Dichloroanthracene	42.0	0.754	0.13	17
3,6-Dichlorodibenzofuran	30.4	0.969	0.14	15
2,3-Dichlorodibenzo-*p*-dioxin	31.2	0.476	0.057	12
2,7-Dichlorodibenzo-*p*-dioxin	17.1	0.320	0.0089	2.8
6,9-Dichloro-2-methoxyacridine	51.0	0.298	0.082	27
1-Fluoronaphthalene	42.8	0.593	0.037	6.3
1,2,3,4,6,7,8-Heptachlorodibenzo-*p*-dioxin	80.4	0.121	0.014	12
1,2,3,4,7,8-Hexachlorodibenzo-*p*-dioxin	15.1	0.0851	0.013	15
1-Iodonaphthalene	45.6	0.378	0.041	11
1,2,3,4,6,7,8,9-Octachlorodibenzofuran	32.0	0.0414	0.011	26
1,2,3,4,6,7,8,9-Octachlorodibenzo-*p*-dioxin	35.6	0.0303	0.0079	26
1,2,3,4,7-Pentachlorodibenzo-*p*-dioxin	32.8	0.246	0.016	6.1
2,3,7,8-Tetrachlorodibenzofuran	22.5	0.212	0.011	5.3
1,2,3,4-Tetrachlorodibenzo-*p*-dioxin	19.4	0.233	0.038	16
1,2,4-Trichlorodibenzo-*p*-dioxin	16.8	0.403	0.022	5.6

[a] RRF = $(A_s C_{is}/A_{is} C_s)$.

TABLE 11. COMPARISON OF THE ESTIMATED LEVELS OF DETECTION BETWEEN FULL MASS ACQUIRE AND SELECTED-ION MONITORING

Compound	Quantitation ion	Acquire detection limit, ng	SIM detection limit, ng
1-Chlorodibenzo-*p*-dioxin	220	9	0.2
2,7-Dichlorodibenzo-*p*-dioxin	254	9	0.2
1,2,4-Trichlorodizenzo-*p*-dioxin	288	10	0.2
1,2,3,4-Tetrachlorodibenzo-*p*-dioxin	322	10	0.2
1,2,3,4,7,8-Hexachlorodibenzo-*p*-dioxin	390	8	0.2
1,2,3,4,6,7,8,9-Octachlorodibenzo-*p*-dioxin	460	40	0.2
1,2,3,4,6,7,8,9-Octachlorodibenzofuran	444	30	0.2

TABLE 12. SUMMARY OF GC/FID DETERMINATIONS OF CANDIDATE POHCs

Compound	Relative retention time[a]	On-column detection limit, ng	Appendix C Reference chromatogram	Appendix C Reference calibration curve
Hydroxylamine, *N*-nitroso-*N*-phenyl-, ammonium salt[b]	0.230	200	C-19	C-20
Propionitrile, 2-hydroxy-	0.262	10	C-45	C-46
Chlorobenzene	0.309	2	C-12	C-13
Styrene	0.360	7	C-45	C-47
Phosphoric acid, trimethyl ester	0.400	10	C-32	C-33
Pyridine, 3-chloromethyl-, hydrochloride	0.420	800	C-19	C-21
m-Dichlorobenzene	0.459	10	C-14	C-15
p-Dichlorobenzene	0.465	10	C-16	C-17
o-Dichlorobenzene	0.486	10	C-16	C-18
o-Anisidine hydrochloride	0.506	200	C-19	C-22
o-Toluidine	0.522	6	C-45	C-48
Tetraethylpyrophosphate	0.554	5	C-56	C-57
2-Oxepanone	0.578	2	C-45	C-49
Hydroquinone	0.583	400	C-19	C-23
o-Anisidine	0.604	7	C-32	C-34
o-Anisidine, 5-methyl-	0.675	4	C-45	C-50
Phosphoric triamide, hexamethyl-	0.686	3	C-40	C-41
Aniline, 2,4,5-trimethyl-	0.689	9	C-32	C-35
o-Toluidine, 5-chloro-	0.692	3	C-62	C-63
Toluene, 2,4-diamino-	0.752	9	C-45	C-51
o-Phenylenediamine, 4-chloro-	0.754	200	C-19	C-24
Phosphoric acid, 1,2-dibromo-2, 2-dichloroethyl dimethyl ester	0.921	4	C-40	C-42
p-Toluidine, *alpha*,*alpha*,*alpha*-trifluoro-2,6-dinitro-*N*,*N*-dipropyl-	0.924	2	C-65	C-66
Phosphoric acid, dimethyl ester, ester with (*E*)-3-hydroxy-*N*,*N*-dimethylcrotonamide	0.925	6	C-25	C-26
Carbamic acid, methyl-,2,3-(dimethyl methylenedioxy)phenyl ester	0.926	2	C-7	C-8
Benzonitrile, 3,5-dibromo-4-hydroxy-	0.934	10	C-45	C-52
o-Anisidine, 5-nitro-	0.963	7	C-32	C-36
Carbamic acid, methyl-2,3-dihydro-2,2-dimethyl-7-benzofuranyl ester	0.966	8	C-1	C-2
1,4-Naphthoquinone, 2,3-dichloro-	1.01	4	C-62	C-64
Biphenyl, 4-nitro	1.03	8	C-32	C-37

(continued)

TABLE 12 (continued)

Compound	Relative retention time[a]	On-column detection limit, ng	Appendix C	
			Reference chromatogram	Reference calibration curve
Carbamic acid, methyl-, 1-naphthyl ester	1.05	8	C-1	C-3
Succinic acid, mercapto-, diethyl ester, *S*-ester with *O*,*O*-dimethyl phosphorodithioate	1.08	30	C-56	C-58
Phosphorothioic acid, *O*,*O*-dimethyl-, *O*-(4-methylthio)-*m*-tolyl) ester	1.09	2	C-40	C-43
S-Triazine, 2,4-dichloro-6-(*o*-chloroanilino)-	1.13	2	C-7	C-9
Phosphoric acid, 2-chloro-1-(2,4-dichlorophenyl) vinyl diethyl ester	1.13	2	C-25	C-27
Heptachlor epoxide	1.14	2	C-65	C-67
Crotonic acid, 3-hydroxy-, *alpha*-methylbenzyl ester, dimethyl phosphate, (*E*)-	1.14	2	C-56	C-59
Aniline, *p*-(phenylazo)-	1.14	7	C-45	C-53
4-Cyclohexene-1,2-dicarboximide, *N*-(trichloromethyl)thio-	1.14	7	C-1	C-4
Carbanilic acid, *m*-chloro-, 4-chloro-2-butynyl ester	1.18	2	C-7	C-1
3-Amino-9-ethyl carbazole	1.20	2	C-45	C-54
Ether, 2,4-dichlorophenyl *p*-nitrophenyl	1.21	2	C-65	C-68
Phosphorothioic acid, *O*,*O*-diethyl *O*-(*p*-(methylsulfinyl) phenyl) ester	1.22	7	C-40	C-44
o-Toluidine, 4-(*o*-tolylazo)-	1.22	7	C-32	C-38
Phosphorodithioic acid, *S*,*S*'-methylene *O*,*O*,*O*',*O*'-tetraethyl ester	1.23	2	C-56	C-60
Pyridine, 2,6-diamino-3-(phenylazo)-, monohydrochloride	1.25	20	C-45	C-55
Phosphorodithioic acid, *S*-(((*p*-chlorophenyl)thio)methyl) *O*,*O*-diethyl ester	1.25	8	C-25	C-28
Aniline, 4,4'-methylenebis(*N*,*N*-dimethyl-	1.26	8	C- 1	C-5
4-Cyclohexene-1,2-dicarboximide, *N*((1,1,2,2,-tetrachloroethyl)thio)-	1.29	6	C-7	C-11

(continued)

TABLE 12 (continued)

Compound	Relative retention time[a]	On-column detection limit, ng	Appendix C	
			Reference chromatogram	Reference calibration curve
Phosphorodithioic acid, *O*,*O*-dimethyl ester, *S*-ester with *N*-(mercaptomethyl) phthalimide	1.31	2	C-25	C-29
Phosphonothioic acid, phenyl-, *O*-(4-bromo-2,5-dichloro-phenyl) *O*-methyl ester	1.34	2	C-25	C-30
Phosphorodithioic acid, *O*,*O*-dimethyl ester, *S*-ester with 3-(mercaptomethyl)1,2,3-benzotriazin-4(3*H*)-one	1.34	20	C-56	C-61
1,3,4-Metheno-1*H*-cyclobuta[cd]pentalene, 1,1a,2,2,3,3a,4,5,5,5a,5b,6-dodecachlorooctahydro-	1.37	9	C-65	C-69
Phosphoric acid, tri-*o*-tolyl ester	1.39	20	C-32	C-39
Coumarin, 3-chloro-7-hydroxy-4-methyl-, *O*-ester with *O*,*O*-diethyl phosphorothioate	1.42	2	C-25	C-31
Hexabromobiphenyl	1.56	20	C-1	C-6

[a] Relative to the retention time of anthracene-d_{10}.

[b] Analyzed as nitrosobenzene.

Also, with each calibration curve is the least-squares plot (a broken line) based on the linear-regression analysis of FID response versus concentration of analyte.

In Table 13, we summarize the GC/MS determination of candidate POHCs. As in Table 12, the retention times are given relative to the internal standard, anthracene-d_{10}. Table 13 also lists the five most abundant mass fragments of each compound and, moreover, specifies the mass of the ion of each compound that was used for the establishment of detection limits and for the generation of calibration curves. The detection limits were estimated from the calibration curves. Typical values of the detection limits were 2.0 to 800 ng.

Reference total-ion chromatograms, calibration curves, and mass spectra are cited in Table 13. The mass spectra are presented in Appendix D along with the reference chromatograms and calibration curves.

In Table 14, we present the results of triplicate GC/FID determinations of all of the candidate POHCs listed in Table 12. The calculated values of the SD and RSD in Table 14 indicate that most GC/FID determinations gave acceptable precision. The RSD ranged from 0.3% to 8%.

In Table 15, we present the results of triplicate GC/MS determinations of all of the POHCs listed in Table 13. In general, the precision of the GC/MS determinations were similar to the GC/FID determinations. The RSDs ranged from 0.4 to 31%.

TABLE 13. SUMMARY OF GC/MS DETERMINATIONS OF CANDIDATE POHCs

Compound	Relative retention time[a]	On-column detection limit, ng	Mol wt	Masses of characteristic EI ions (relative abundance)					Appendix B		
				1	2	3	4	5	Reference chromatogram	Reference mass spectrum	Reference calibration curve
Propionitrile, 2-hydroxy-	0.208	80	71	56(100)	42(33)	43(16)	70(9)	44(7)	D-75	D-76	D-77
Chlorobenzene	0.230	10	112	112(100)	77(43)	114(32)	51(8)	113(7)	D-22	D-23	D-24
Styrene	0.269	10	104	104(100)	103(49)	78(34)	77(17)	51(11)	D-89	D-90	D-91
Hydroxylamine, N-nitroso-N-phenyl-, ammonium salt[b]	0.304	200	156	77(100)	107(90)	51(50)	50(15)	78(6)	D-75	D-78	D-79
Phosphoric acid, trimethyl ester	0.326	90	140	110(100)	109(30)	79(29)	95(25)	80(23)	D-51	D-52	D-53
Pyridine, 3-chloromethyl-, hydrochloride	0.363	700	163	92(100)	127(46)	65(35)	129(18)	128(10)	D-80	D-81	D-82
m-Dichlorobenzene	0.405	10	146	146(100)	148(63)	111(24)	150(11)	75(10)	D-25	D-26	D-27
p-Dichlorobenzene	0.413	10	146	146(100)	148(67)	111(27)	75(12)	113(10)	D-28	D-29	D-30
o-Dichlorobenzene	0.434	10	146	146(100)	148(63)	111(29)	75(13)	113(13)	D-28	D-31	D-32
o-Anisidine hydrochloride	0.460	800	159	108(100)	80(77)	123(72)	53(18)	65(14)	D-80	D-83	D-84
o-Toluidine	0.462	6	107	106(100)	107(74)	77(16)	79(14)	89(7)	D-89	D-92	D-93
2-Oxepanone	0.463	20	114	55(100)	42(58)	56(42)	84(36)	114(32)	D-89	D-94	D-95
Tetraethylpyrophosphate	0.528	20	290	99(100)	155(70)	127(51)	81(49)	109(35)	D-108	D-109	D-110
Hydroquinone	0.557	800	110	110(100)	81(25)	55(21)	108(17)	82(16)	D-80	D-85	D-86
o-Anisidine	0.559	4	123	123(100)	108(99)	80(63)	53(12)	52(10)	D-51	D-54	D-55
o-Anisidine, 5-methyl-	0.642	20	137	122(100)	137(65)	94(46)	77(22)	93(10)	D-89	D-96	D-97
Aniline, 2,4,5-trimethyl-	0.653	10	135	120(100)	135(84)	134(59)	91(17)	65(10)	D-51	D-56	D-57
Phosphoric triamide, hexamethyl-	0.669	20	179	135(100)	44(56)	179(47)	45(35)	136(26)	D-66	D-67	D-68
o-Toluidine, 5-chloro-	0.674	6	141	141(100)	106(79)	140(75)	143(37)	142(32)	D-119	D-120	D-121
Toluene, 2,4-diamino-	0.728	20	122	121(100)	122(99)	94(22)	77(14)	104(13)	D-89	D-98	D-99
o-Phenylenediamine, 4-chloro-	0.734	200	142	142(100)	144(30)	80(29)	141(28)	114(24)	D-80	D-87	D-88
Phosphoric acid, 1,2-dibromo-2,2-dichloroethyl dimethyl ester	0.919	4	378	109(100)	145(45)	79(22)	147(14)	47(9)	D-66	D-69	D-70
Phosphoric acid, dimethyl ester, ester with (E)-3-hydroxy-N,N-dimethylcrotonamide	0.928	30	237	127(100)	67(26)	72(9)	193(8)	44(7)	D-38	D-39	D-40
Carbamic acid, methyl-, 2,3-(dimethylmethylenedioxy)phenyl ester	0.929	8	223	151(100)	126(42)	166(35)	223(15)	51(15)	D-13	D-14	D-15
p-Toluidine, alpha,alpha,alpha-trifluoro-2,6-dinitro-N,N-dipropyl-	0.931	9	335	306(100)	264(39)	335(19)	43(16)	307(12)	D-127	D-128	D-129

(continued)

TABLE 13 (continued)

Compound	Relative retention time[a]	On-column detection limit, ng	Mol wt	Masses of characteristic EI ions (relative abundance) 1	2	3	4	5	Appendix B Reference chromatogram	Appendix B Reference mass spectrum	Appendix B Reference calibration curve
Benzonitrile, 3,5-dibromo-4-hydroxy-	0.933	30	275	88(100)	277(78)	275(45)	279(40)	62(27)	D-89	D-100	D-101
o-Anisidine, 5-nitro-	0.968	20	168	168(100)	153(36)	79(24)	52(24)	122(23)	D-51	D-58	D-59
Carbamic acid, methyl-, 2,3-dihydro-2,2-dimethyl-7-benzofuranyl ester	0.970	8	221	164(100)	149(55)	122(14)	131(14)	123(13)	D-1	D-2	D-3
p-Toluidine, N-(2-chloroethyl)-2,6-dinitro-N-propyl-alpha,alpha,alpha-trifluoro-	1.00	8	355	63(100)	306(92)	326(62)	264(59)	43(55)	D-124	D-125	D-126
1,4-Naphthoquinone, 2,3-dichloro-	1.01	20	226	191(100)	226(96)	163(66)	228(58)	135(40)	D-119	D-122	D-123
Carbamic acid, methyl-, 4-dimethylamino-3,4-xylyl ester	1.02	7	222	165(100)	150(92)	164(85)	134(60)	77(29)	D-10	D-11	D-12
Biphenyl, 4-nitro	1.04	2	199	199(100)	152(94)	153(26)	151(26)	141(20)	D-51	D-60	D-61
Carbamic acid, methyl-, 1-naphthyl ester	1.06	2	201	144(100)	115(36)	116(22)	145(11)	57(7)	D-1	D-4	D-5
Succinic acid, mercapto-, diethyl ester, S-ester with O,O-dimethyl phosphorodithioate	1.10	10	330	127(100)	93(92)	125(89)	173(87)	158(49)	D-108	D-111	D-112
Phosphorothioic acid, O,O-dimethyl-, O-(4-methylthio)-m-tolyl) ester	1.11	7	278	278(100)	125(72)	109(68)	169(38)	79(35)	D-66	D-71	D-72
Heptachlor epoxide	1.14	9	386	81(100)	353(66)	355(47)	351(36)	237(25)	D-127	D-130	D-131
S-Triazine, 2,4-dichloro-6-(o-chloroanilino)-	1.14	8	274	239(100)	241(64)	178(22)	143(16)	240(12)	D-13	D-16	D-17
4-Cyclohexene-1,2-dicarboximide, N-(trichloromethyl)thio-	1.15	2	229	79(100)	149(24)	77(23)	80(22)	119(20)	D-1	D-6	D-7
Phosphoric acid, 2-chloro-1-(2,4-dichloro phenyl)vinyl diethyl ester	1.15	20	358	267(100)	269(65)	323(53)	325(34)	81(27)	D-38	D-41	D-42
Crotonic acid, 3-hydroxy-, alpha-methylbenzyl ester, dimethyl phosphate, (E)-	1.16	2	314	127(100)	105(44)	193(26)	166(14)	104(13)	D-108	D-113	D-114
Aniline, p-(phenylazo)-	1.17	20	197	92(100)	197(39)	65(32)	120(30)	77(23)	D-89	D-102	D-103
Carbanilic acid, m-chloro-, 4-chloro-2-butynyl ester	1.20	20	257	222(100)	153(95)	51(58)	127(45)	224(36)	D-13	D-18	D-19
3-Amino-9-ethyl carbazole	1.23	20	210	195(100)	210(83)	181(18)	196(15)	211(12)	D-89	D-104	D-105

(continued)

TABLE 13 (continued)

Compound	Relative retention time[a]	On-column detection limit, ng	Mol wt	Masses of characteristic EI ions (relative abundance) 1	2	3	4	5	Appendix B Reference chromatogram	Reference mass spectrum	Reference calibration curve
Phosphorothioic acid, O,O-diethyl O-(p-(methylsulfinyl) phenyl) ester	1.24	20	308	97(100)	293(99)	125(75)	141(74)	109(49)	D-66	D-73	D-74
Phosphorodithioic acid, S,S'-methylene O,O,O',O'-tetraethyl ester	1.26	8	384	231(100)	97(72)	153(61)	125(41)	121(39)	D-108	D-115	D-116
o-Toluidine, 4-(o-tolylazo)-	1.26	20	225	106(100)	225(82)	91(20)	134(19)	224(16)	D-51	D-62	D-63
Phosphorodithioic acid, S-(((p-chlorophenyl) thio)methyl) O,O-diethyl ester	1.28	20	342	157(100)	97(59)	153(49)	121(49)	125(41)	D-38	D-43	D-44
Aniline, 4,4'-methylenebis(N,N-dimethyl-	1.28	8	254	254(100)	253(79)	134(60)	126(57)	118(47)	D-1	D-8	D-9
Pyridine, 2,6-diamino-3-(phenylazo)-, monohydrochloride	1.29	30	249	108(100)	81(92)	54(50)	213(47)	77(41)	D-89	D-106	D-107
4-Cyclohexene-1,2-dicarboximide, N-((1,1,2,2-tetrachloroethyl)thio)-	1.30	10	347	79(100)	313(52)	311(51)	349(40)	312(34)	D-13	D-20	D-21
Phosphorodithioic acid, O,O-dimethyl ester, S-ester with N-(mercaptomethyl)phthalimide	1.34	4	317	60(100)	161(10)	77(7)	93(6)	76(6)	D-38	D-45	D-46
Phosphorodithioic acid, O,O-dimethyl ester, S-ester with 3-(mercaptomethyl)-1,2,3-benzotriazin-4(3H)-one	1.38	20	317	60(100)	177(68)	132(64)	92(21)	76(16)	D-108	D-117	D-118
Phosphonothioic acid, phenyl-, O-(4-bromo-2,5-dichlorophenyl) O-methyl ester	1.38	10	410	171(100)	177(52)	155(34)	63(32)	377(21)	D-38	D-47	D-48
Ether, 2,4-dichlorophenyl p-nitrophenyl	1.38	20	283	283(100)	202(82)	50(68)	63(64)	285(60)	D-127	D-132	D-133
1,3,4-Metheno-1H-cyclobuta[cd]pentalene, 1,1a,2,2,3,3a,4,5,5,5a,5b,6-dodecachlorooctahydro-	1.38	9	540	272(100)	274(78)	237(51)	270(48)	239(35)	D-33	D-34	D-35
Coumarin, 3-chloro-7-hydroxy-4-methyl-, O-ester with O,O-diethyl phosphorothioate	1.44	10	362	362(100)	226(62)	97(49)	109(46)	210(41)	D-38	D-49	D-50
Phosphoric acid, tri-o-tolyl ester	1.50	30	368	368(100)	367(54)	91(27)	165(21)	369(21)	D-51	D-64	D-65
Hexabromobiphenyl	1.53	80	622	308(100)	467(77)	234(68)	465(58)	306(52)	D-33	D-36	D-37

[a] Relative to the retention time of anthracene-d_{10}.

[b] Analyzed as nitrosobenzene.

TABEL 14. PRECISION OF GC/FID DETERMINATIONS

Compound	Quantity injected, ng	RRF[a] Mean	SD	RSD, %
3-Amino-9-ethyl carbazole	36.0	0.383	0.010	2.7
Aniline, 4,4'-methylenebis(*N*,*N*-dimethyl-	41.2	0.614	0.044	7.2
Aniline, *p*-(phenylazo)-	35.2	0.567	0.017	3.0
Aniline, 2,4,5-trimethyl-	48.4	1.07	0.026	2.4
o-Anisidine	36.6	0.821	0.026	3.2
o-Anisidine hydrochloride	4080	0.0521	0.00030	0.59
o-Anisidine, 5-methyl-	38.4	0.791	0.016	2.0
o-Anisidine, 5-nitro-	36.6	0.373	0.0011	2.9
Benzonitrile, 3,5-dibromo-4-hydroxy-	51.0	0.199	0.0085	4.3
Biphenyl, 4-nitro	38.8	0.724	0.0074	1.0
Carbamic acid, methyl-, 2,3-dihydro-2,2-dimethyl-7-benzofuranyl ester	40.6	0.549	0.0069	1.2
Carbamic acid, methyl-, 2,3-(dimethylmethylenedioxy) phenyl ester	40.6	0.443	0.012	2.8
Carbamic acid, methyl-, 1-naphthyl ester	38.2	0.582	0.020	3.4
Carbanilic acid, *m*-chloro-, 4-chloro-2-butynyl ester	36.2	0.297	0.0010	0.34
Chlorobenzene	55.4	0.952	0.0035	0.37
Coumarin, 3-chloro-7-hydroxy-4-methyl-, *O*-ester with *O*,*O*-diethyl phosphorothioate	38.0	0.201	0.010	5.1
Crotonic acid, 3-hydroxy-, *alpha*-methylbenzyl ester, dimethyl phosphate, (*E*)-	47.2	0.334	0.013	3.8
4-Cyclohexene-1,2-dicarboximide, *N*((1,1,2,2,-tetrachloroethyl)thio)-	56.6	0.210	0.0055	2.6
4-Cyclohexene-1,2-dicarboximide, *N*-(trichloromethyl)thio-	37.0	0.238	0.016	6.5
m-Dichlorobenzene	51.6	0.570	0.0085	1.5
o-Dichlorobenzene	53.6	0.627	0.0080	1.3
p-Dichlorobenzene	41.6	0.634	0.010	1.6
Ether, 2,4-dichlorophenyl *p*-nitrophenyl	35.6	0.379	0.017	4.6
Heptachlor epoxide	45.8	0.345	0.0028	0.80
Hexabromobiphenyl	78.2	0.0807	0.0042	5.2
Hydroquinone	3960	0.0499	0.00040	0.89
Hydroxylamine, *N*-nitroso-*N*-phenyl-, ammonium salt[b]	3910	0.0253	0.0012	4.7

(continued)

TABLE 14 (continued)

Compound	Quantity injected, ng	RRF[a]		
		Mean	SD	RSD, %
1,3,4-Metheno-1*H*-cyclobuta[cd]pentalene, 1,1a,2,2,3,3a,4,5,5,5a,5b,6-dodecachlorooctahydro-	44.4	0.235	0.0049	2.1
1,4-Naphthoquinone, 2,3-dichloro-	40.6	0.471	0.015	3.1
2-Oxepanone	35.2	0.662	0.012	1.8
o-Phenylenediamine, 4-chloro-	3920	0.0458	0.0014	3.0
Phosphonothioic acid, phenyl-, *O*-(4-bromo-2,5-dichloro-phenyl) *O*-methyl ester	33.2	0.329	0.012	3.7
Phosphoric acid, 2-chloro-1-(2,4-dichlorophenyl) vinyl diethyl ester	41.4	0.317	0.0070	2.2
Phosphoric acid, 1,2-dibromo-2,2-dichloroethyl dimethyl ester	44.4	0.105	0.00045	0.43
Phosphoric acid, dimethyl ester, ester with (*E*)-3-hydroxy-*N*,*N*-dimethylcrotonamide	59.6	0.258	0.0063	2.4
Phosphoric acid, trimethyl ester	71.2	0.189	0.0049	2.6
Phosphoric acid, tri-*o*-tolyl ester	76.0	0.0890	0.0032	3.6
Phosphoric triamide, hexamethyl-	62.0	0.410	0.013	3.1
Phosphorodithioic acid, *S*-(((*p*-chlorophenyl) thio)methyl) *O*,*O*-diethyl ester	37.6	0.364	0.0084	2.3
Phosphorodithioic acid, *O*,*O*-dimethyl ester, *S*-ester with 3-(mercaptomethyl) 1,2,3-benzotriazin-4(3*H*)-one	36.2	0.196	0.016	8.3
Phosphorodithioic acid, *O*,*O*-dimethyl ester, *S*-ester with *N*-(mercaptomethyl) phthalimide	43.6	0.269	0.011	4.2
Phosphorodithioic acid, *S*,*S*'-methylene *O*,*O*,*O*',*O*'-tetraethyl ester	38.0	0.239	0.0072	3.0
Phosphorothioic acid, *O*,*O*-diethyl *O*-(*p*-(methylsulfinyl) phenyl) ester	34.0	0.235	0.014	6.1
Phosphorothioic acid, *O*,*O*-dimethyl-, *O*-(4-methylthio)-*m*-tolyl) ester	34.8	0.423	0.0052	1.2
Propionitrile, 2-hydroxy-	51.4	0.262	0.012	4.5
Pyridine, 3-chloromethyl-, hydrochloride	3680	0.0356	0.0011	3.2
Pyridine, 2,6-diamino-3-(phenylazo)-, monohydrochloride	31.4	0.209	0.0042	2.0
Styrene	34.4	1.23	0.041	3.4
Succinic acid, mercapto-, diethyl ester, *S*-ester with *O*,*O*-dimethyl phosphorodithioate	58.8	0.248	0.0012	0.46

(continued)

TABLE 14 (continued)

Compound	Quantity injected, ng	RRF[a] Mean	RRF[a] SD	RRF[a] RSD, %
Tetraethylpyrophosphate	47.0	0.105	0.00052	0.49
Toluene, 2,4-diamino-	47.0	0.438	0.0039	0.90
o-Toluidine	61.6	1.08	0.020	1.8
o-Toluidine, 5-chloro-	32.2	0.723	0.021	2.8
o-Toluidine, 4-(*o*-tolylazo)-	35.0	0.518	0.0080	1.5
p-Toluidine, *alpha*,*alpha*,*alpha*-trifluoro-2,6-dinitro-*N*,*N*-dipropyl-	44.8	0.521	0.010	2.0
S-Triazine, 2,4-dichloro-6-(*o*-chloroanilino)-	38.6	0.310	0.0021	0.67

[a] RRF = $(A_sC_{is}/A_{is}C_s)$

[b] Analyzed as nitrosobenzene.

TABLE 15. PRECISION OF GC/MS DETERMINATIONS

Compound	Quantity injected, ng	RRF[a] Mean	RRF[a] SD	RRF[a] RSD, %
3-Amino-9-ethyl carbazole	36.0	0.340	0.019	5.5
Aniline, 4,4'-methylenebis(*N*,*N*-dimethyl)-	41.2	0.449	0.013	2.9
Aniline, *p*-(phenylazo)-	35.2	0.668	0.053	7.9
Aniline, 2,4,5-trimethyl-	48.4	0.441	0.025	5.7
o-Anisidine	36.6	0.294	0.046	16
o-Anisidine hydrochloride	4080	0.029	0.0017	5.9
o-Anisidine, 5-methyl-	38.4	0.586	0.083	14
o-Anisidine, 5-nitro-	36.6	0.159	0.0085	5.3
Benzonitrile, 3,5-dibromo-4-hydroxy-	51.0	0.349	0.034	9.7
Biphenyl, 4-nitro	38.8	0.351	0.021	5.9
Carbamic acid, methyl-, 2,3-dihydro-2,2-dimethyl-7-benzofuranyl ester	40.6	0.454	0.026	5.7
Carbamic acid, methyl-, 4-dimethylamino-3,4-xylyl ester	34.4	0.351	0.11	31
Carbamic acid, methyl-, 2,3-(dimethylmethylenedioxy)phenyl ester	40.6	0.541	0.086	16
Carbamic acid, methyl-, 1-naphthyl ester	38.2	0.696	0.023	3.2
Carbanilic acid, *m*-chloro-, 4-chloro-2-butynyl ester	36.2	0.0853	0.022	26
Chlorobenzene	55.3	0.333	0.014	4.2
Coumarin, 3-chloro-7-hydroxy-4-methyl-, *O*-ester with *O*,*O*-diethyl phosphorothioate	38.0	0.0733	0.0081	11
Crotonic acid, 3-hydroxy-, *alpha*-methylbenzyl ester, dimethyl phosphate, (*E*)-	47.2	0.530	0.054	10
4-Cyclohexene-1,2-dicarboximide, *N*-((1,1,2,2-tetrachloroethyl)thio)-	56.6	0.293	0.045	15
4-Cyclohexene-1,2-dicarboximide, *N*-(trichloromethyl)thio-	37.0	0.323	0.040	12
m-Dichlorobenzene	51.6	0.552	0.018	3.5
o-Dichlorobenzene	41.7	0.660	0.029	4.4
p-Dichlorobenzene	52.2	0.441	0.018	4.0
Ether, 2,4-dichlorophenyl *p*-nitrophenyl	35.6	0.182	0.015	8.2
Heptachlor epoxide	45.8	0.102	0.016	16
Hexabromobiphenyl	78.0	0.0314	0.0039	12
Hydroquinone	3960	0.0321	0.0019	6.0
Hydroxylamine, *N*-nitroso-*N*-phenyl-, ammonium salt[b]	777	0.235	0.050	21
1,3,4-Metheno-1*H*-cyclobuta[cd]pentalene, 1,1a,2,2,3,3a,4,5,5,5a,5b,6-dodecachlorooctahydro-	44.4	0.442	0.11	24

(continued)

TABLE 15 (continued)

Compound	Quantity injected, ng	RRF^{a}		
		Mean	SD	RSD, %
1,4-Naphthoquinone, 2,3-dichloro-	40.6	0.150	0.0041	2.7
2-Oxepanone	35.2	0.371	0.0041	1.1
o-Phenylenediamine, 4-chloro-	3920	0.0215	0.00038	1.8
Phosphonothioic acid, phenyl-, *O*-(4-bromo-2,5-dichloro-phenyl) *O*-methyl ester	33.2	0.293	0.012	3.9
Phosphoric acid, 2-chloro-1-(2,4-dichlorophenyl) vinyl diethyl ester	41.4	0.182	0.0044	2.4
Phosphoric acid, 1,2-dibromo-2,2-dichloroethyl dimethyl ester	44.4	0.282	0.015	5.2
Phosphoric acid, dimethyl ester, ester with (*E*)-3-hydroxy-*N*,*N*-dimethylcrotonamide	59.6	0.589	0.015	2.6
Phosphoric acid, trimethyl ester	71.2	0.483	0.11	22
Phosphoric acid, tri-*o*-tolyl ester	76.0	0.0513	0.0092	18
Phosphoric triamide, hexamethyl-	62.0	0.524	0.084	16
Phosphorodithioic acid, *S*-(((*p*-chlorophenyl) thio)methyl) *O*,*O*-diethyl ester	37.6	0.227	0.0099	4.3
Phosphorodithioic acid, *O*,*O*-dimethyl ester, *S*-ester with 3-(mercaptomethyl)-1,2,3-benzotriazin-4(3*H*)-one	36.2	0.229	0.027	12
Phosphorodithioic acid, *O*,*O*-dimethyl ester, *S*-ester with *N*-(mercaptomethyl)phthalimide	43.6	0.751	0.020	2.7
Phosphorodithioic acid, *S*,*S*'-methylene *O*,*O*,*O*',*O*'-tetraethyl ester	38.0	0.370	0.035	9.6
Phosphorothioic acid, *O*,*O*-diethyl *O*-(*p*-(methylsulfinyl) phenyl) ester	34.0	0.0562	0.0097	17
Phosphorothioic acid, *O*,*O*-dimethyl-, *O*-(4-methylthio)-*m*-tolyl) ester	34.8	0.330	0.0086	2.6
Propionitrile, 2-hydroxy-	834	0.357	0.021	5.9
Pyridine, 3-chloromethyl-, hydrochloride	3680	0.0320	0.00012	0.38
Pyridine, 2,6-diamino-3-(phenylazo)-, monohydrochloride	31.4	0.107	0.018	17
Styrene	34.4	0.740	0.058	7.9
Succinic acid, mercapto-, diethyl ester, *S*-ester with *O*,*O*-dimethyl phosphorodithioate	58.8	0.198	0.017	8.4
Tetraethylpyrophosphate	47.0	0.128	0.0051	4.0
Toluene, 2,4-diamino-	47.0	0.317	0.017	5.3

(continued)

TABLE 15 (continued)

Compound	Quantity injected, ng	RRF[a] Mean	SD	RSD, %
o-Toluidine	61.6	1.80	0.23	13
o-Toluidine, 5-chloro-	32.2	0.249	0.021	8.4
p-Toluidine, *N*-(2-chloroethyl)-2,6-dinitro-*N*-propyl-*alpha*,*alpha*,*alpha*-trifluoro-	39.0	0.128	0.0043	3.3
o-Toluidine, 4-(*o*-tolylazo)-	35.0	0.351	0.011	3.1
p-Toluidine, *alpha*,*alpha*,*alpha*-trifluoro-2,6-dinitro-*N*,*N*-dipropyl-	44.8	0.338	0.10	30
S-Triazine, 2,4-dichloro-6-(*o*-chloroanilino)-	38.6	0.376	0.064	17

[a] Relative to the retention time of anthracene-d_{10}.

[b] Analyzed as nitrosobenzene.

SECTION 7

QUALITY-ASSURANCE SUMMARY

The work that was completed at SoRI and presented in this report was performed under SoRI Quality Assurance Project Plan, entitled "PIC Method Analysis" for EPA Contract 68-02-3696, Work Assignment 004, March 15, 1984. This project mainly addresses general research associated with analytical method development. Although the project includes environmentally related measurement activities, SoRI was not involved in field sampling. Thus, sampling procedures were considered as non-applicable in the systematic review of this project. The major efforts of SoRI were to evaluate and, when necessary, modify GC/FID and GC/MS generalized analysis procedures for candidate PICs and POHCs. This task was accomplished with readily available compounds of known purities.

An internal audit of this project revealed no serious deficiencies. The GC/MS was not calibrated daily with DFTPP as specified in the QAPP. Because only neat solutions of PICs and POHCs were submitted to GC/FID and GC/MS analyses, recovery data for spiked-matrixes were not available. However, the overall precision of the GC/FID determinations was <9.3% RSD, and for the GC/MS determinations, <30% RSD. Typical ranges (50% of the compounds) for PICs were 0.65 to 2.3% RSD by GC/FID and 7.0 to 15% RSD by GC/MS. Typical ranges for POHCs were 1.5 to 3.4% RSD by GC/FID and 5.2 to 12% by GC/MS.

The relative retention times, on-column detection limits, chromatograms, calibration curves, relative ion abundances, spectra, and relative standard deviations in this report will be a guide for other anlaytical laboratories. Because of subtle differences among laboratories and analytical instrumentation, analysts who participate in similar studies should generate data that will establish their own criteria for the analysis of PICs and POHCs.

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APPENDIX A

CHROMATOGRAMS AND CALIBRATION CURVES FOR GC/FID DETERMINATIONS

Index

Compound	Reference chromatogram Figure No.	Reference calibration curve Figure No.
1-Amino-4-chloronaphthalene	A-34	A-36
1-Amino-4-bromonaphthalene	A-34	A-38
5-Bromoacenaphthene	A-22	A-26
9-Bromoanthracene	A-22	A-30
2-Bromofluorene	A-22	A-28
9-Bromofluorene	A-34	A-37
1-Bromo-2-methylnaphthalene	A-1	A-3
1-Bromo-4-methylnaphthalene	A-22	A-25
2-(Bromomethyl)naphthalene	A-1	A-4
2-Bromonaphthalene	A-1	A-2
9-Bromophenanthrene	A-1	A-6
3-Bromoquinoline	A-22	A-24
1-Chloroanthracene	A-1	A-5
2-Chloroanthracene	A-34	A-39
9-Chloroanthracene	A-22	A-29
1-Chlorodibenzo-*p*-dioxin	A-9	A-10
2-Chlorodibenzo-*p*-dioxin	A-17	A-18
9-(Chloromethyl)anthracene	A-34	A-40
1-Chloro-8-nitronaphthalene	A-22	A-27
9,10-Dibromoanthracene	A-34	A-41
3,6-Dibromocarbazole	A-22	A-31
9,10-Dichloroanthracene	A-1	A-7
3,6-Dichlorodibenzofuran	A-20	A-21
2,3-Dichlorodibenzo-*p*-dioxin	A-17	A-19
2,7-Dichlorodibenzo-*p*-dioxin	A-9	A-11
6,9-Dichloro-2-methoxyacridine	A-1	A-8
1-Fluoronaphthalene	A-22	A-23
1,2,3,4,6,7,8-Heptachlorodibenzo-*p*-dioxin	A-32	A-33
1,2,3,4,7,8-Hexachlorodibenzo-*p*-dioxin	A-9	A-14
1-Iodonaphthalene	A-34	A-35
1,2,3,4,6,7,8,9-Octachlorodibenzofuran	A-9	A-16
1,2,3,4,6,7,8,9-Octachlorodibenzo-*p*-dioxin	A-9	A-15
1,2,3,4,7-Pentachlorodibenzo-*p*-dioxin	A-42	A-43
2,3,7, 8-Tetrachlorodibenzofuran	A-44	A-45
1,2,3,4-Tetrachlorodibenzo-*p*-dioxin	A-9	A-13
1,2,4-Trichlorodibenzo-*p*-dioxin	A-9	A-12

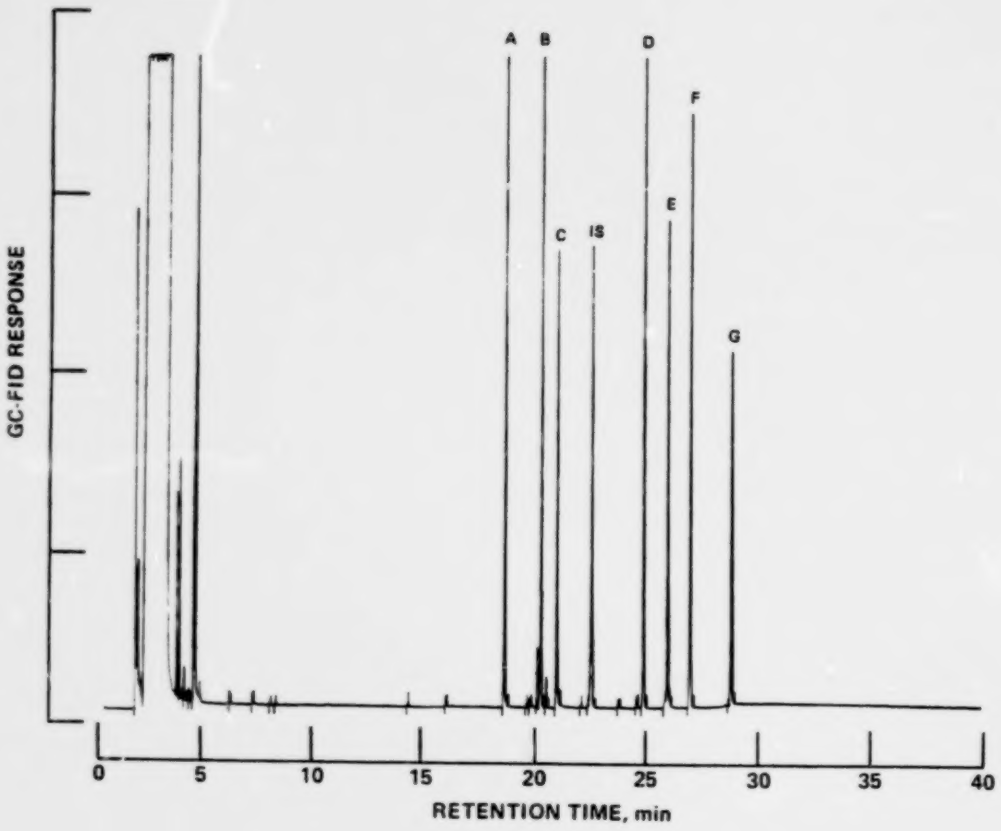


Figure A-1. Chromatogram by GC/FID for

(A) 2-bromonaphthalene(t_R = 18.5 min),
(B) 1-bromo-2-methylnaphthalene(t_R = 20.2 min),
(C) 2-(bromomethyl)naphthalene(t_R = 20.9 min),
(D) 1-chloroanthracene(t_R = 25.0 min),
(E) 9-bromophenanthrene(t_R = 26.0 min),
(F) 9,10-dichloroanthracene(t_R = 27.1 min), and
(G) 6,9-dichloro-2-methoxyacridine(t_R = 28.9 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

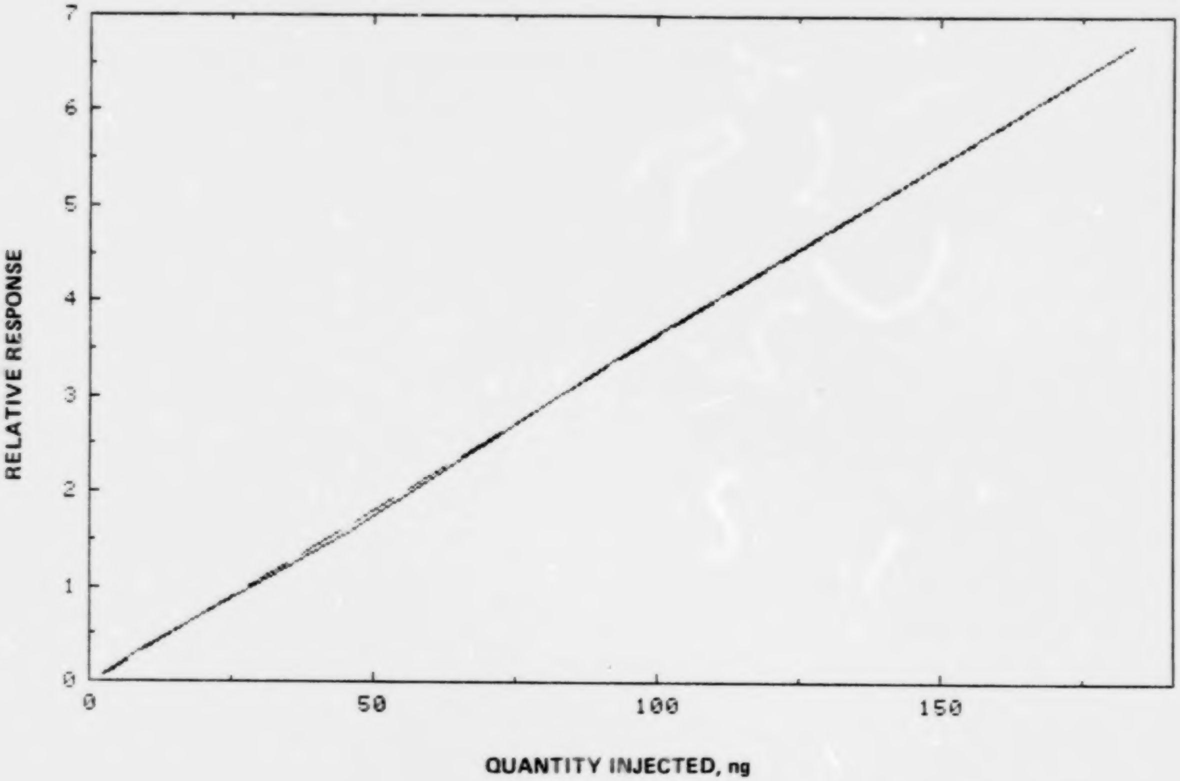


Figure A-2. Calibration curve for the determination of 2-bromonaphthalene by GC/FID.

Range: 4.6 - 180 ng
Correlation coefficient: 0.999
Slope: 3.7×10^{-2}
y-Intercept: -1.9×10^{-2}

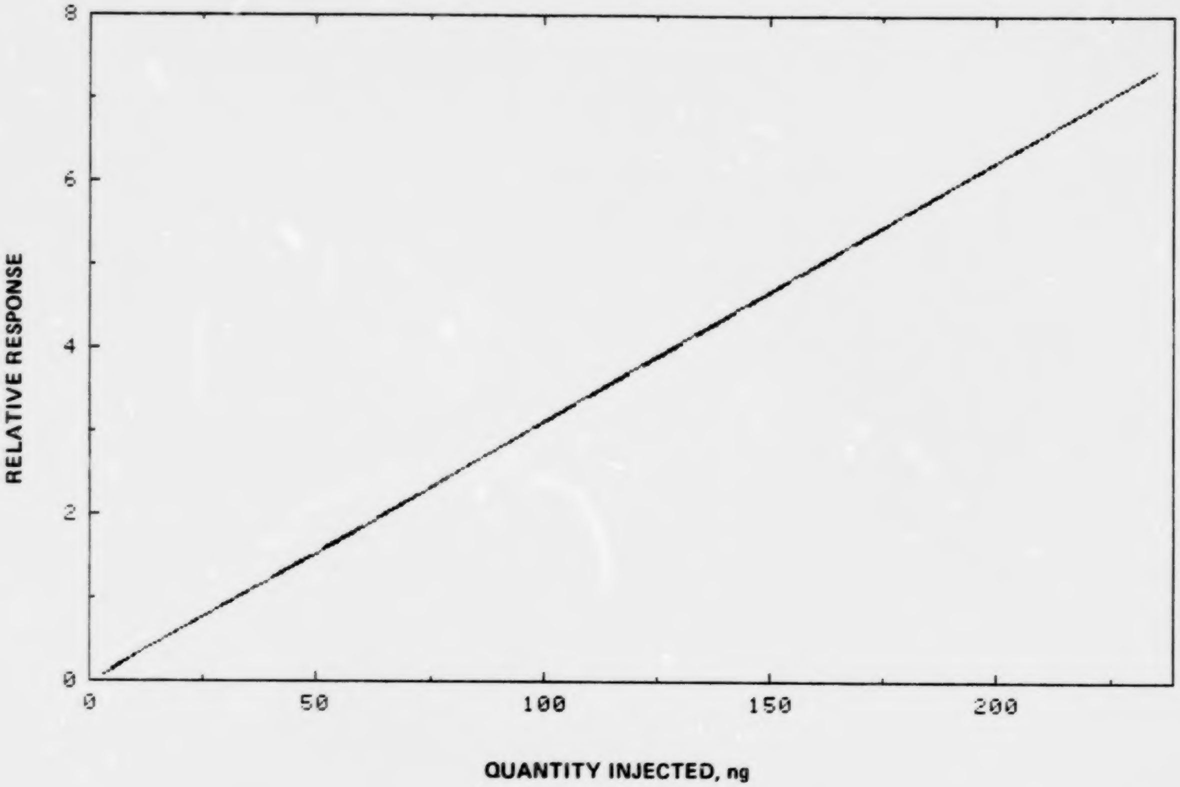


Figure A-3. Calibration curve for the determination of 1-bromo-2-methylnaphthalene by GC/FID.

Range: 3.0 - 240 ng
Correlation coefficient: 0.999
Slope: 3.1×10^{-2}
y-Intercept: -1.0×10^{-2}

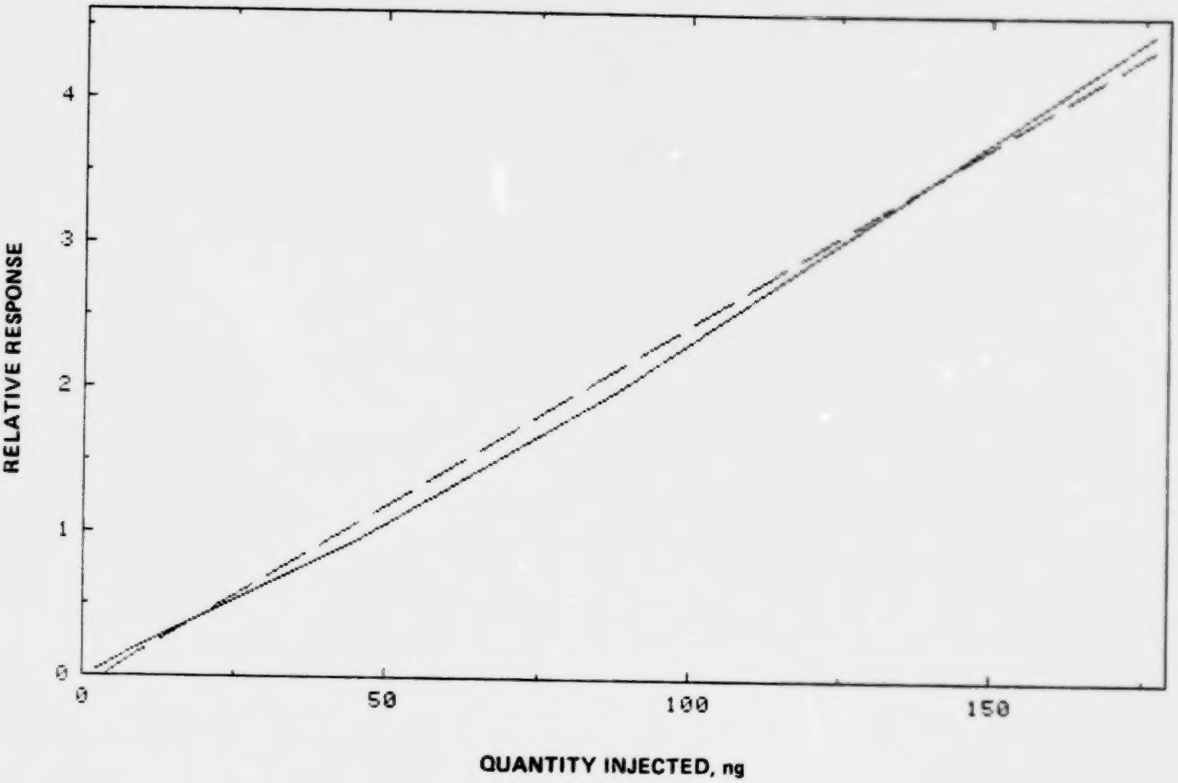


Figure A-4. Calibration curve for the determination of 2-(bromomethyl)naphthalene by GC/FID.

Range: 8.8 - 180 ng
Correlation coefficient: 0.998
Slope: 2.5×10^{-2}
y-Intercept: -7.7×10^{-2}

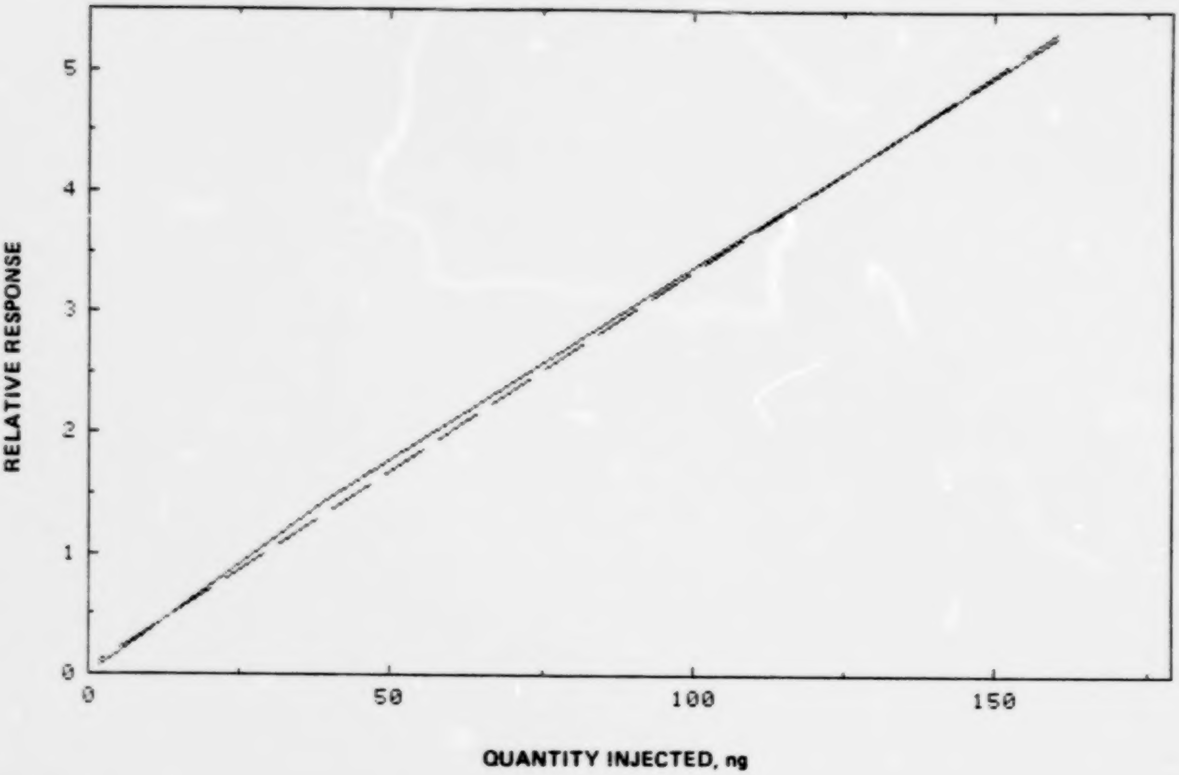


Figure A-5. Calibration curve for the determination of 1-chloroanthracene by GC/FID.

Range: 4.0 - 160 ng
Correlation coefficient: 0.999
Slope: 3.3 x 10^{-2}
y-Intercept: 4.3 x 10^{-2}

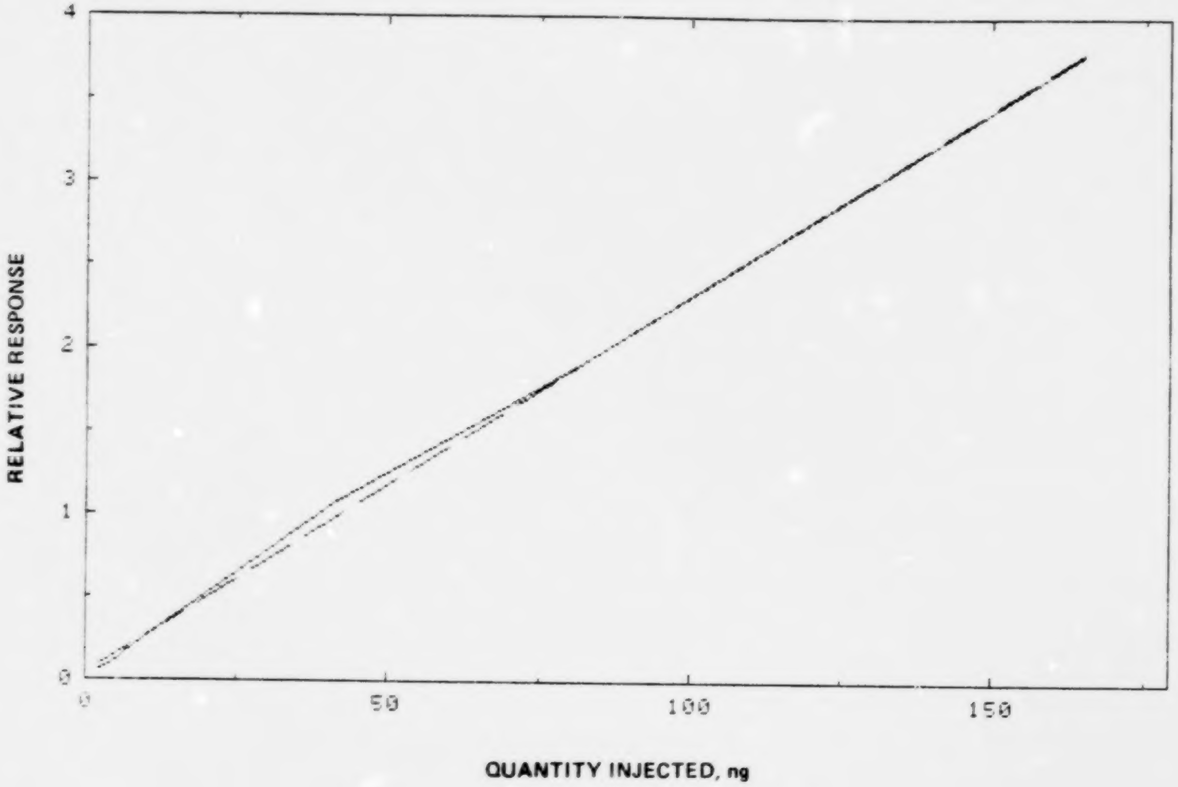


Figure A-6. Calibration curve for the determination of 9-bromophenanthrene by GC/FID.

Range: 4.1 - 160 ng
Correlation coefficient: 0.999
Slope: 2.3×10^{-2}
y-Intercept: 3.9×10^{-2}

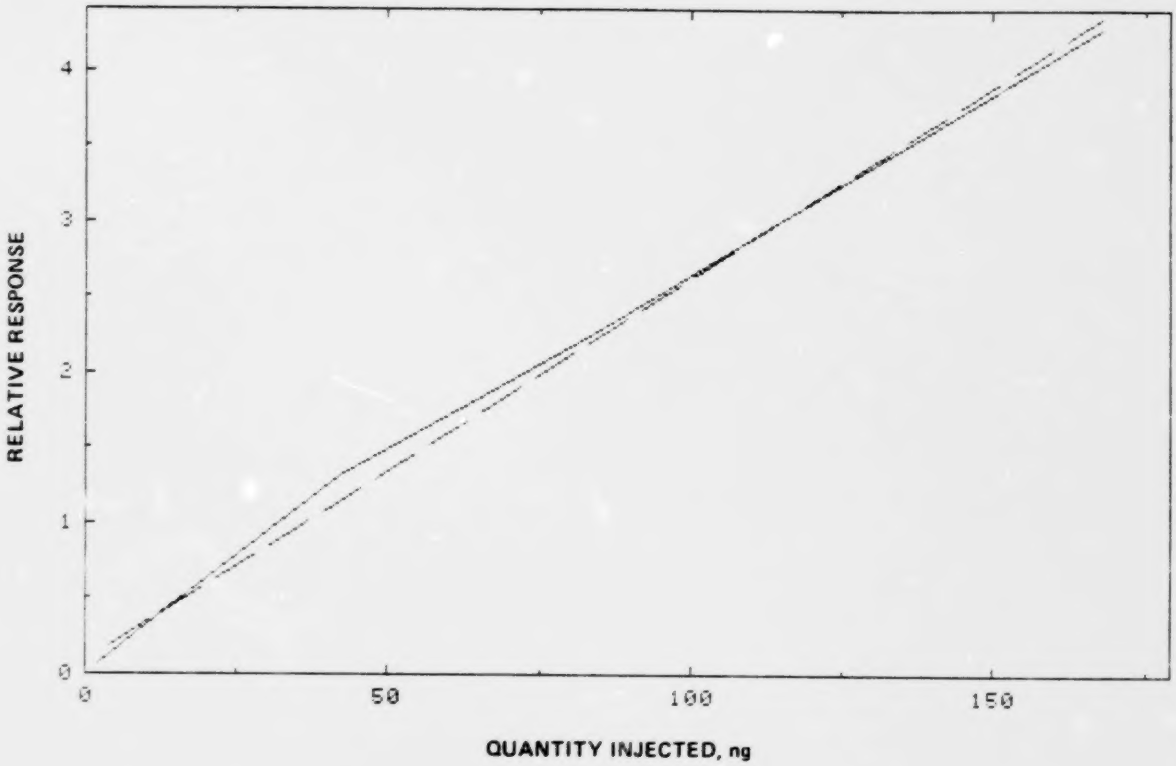


Figure A-7. Calibration curve for the determination of 9,10-dichloroanthracene by GC/FID.

Range: 4.2 - 170 ng
Correlation coefficient: 0.998
Slope: 2.5 x 10^{-2}
y-Intercept: 8.4 x 10^{-2}

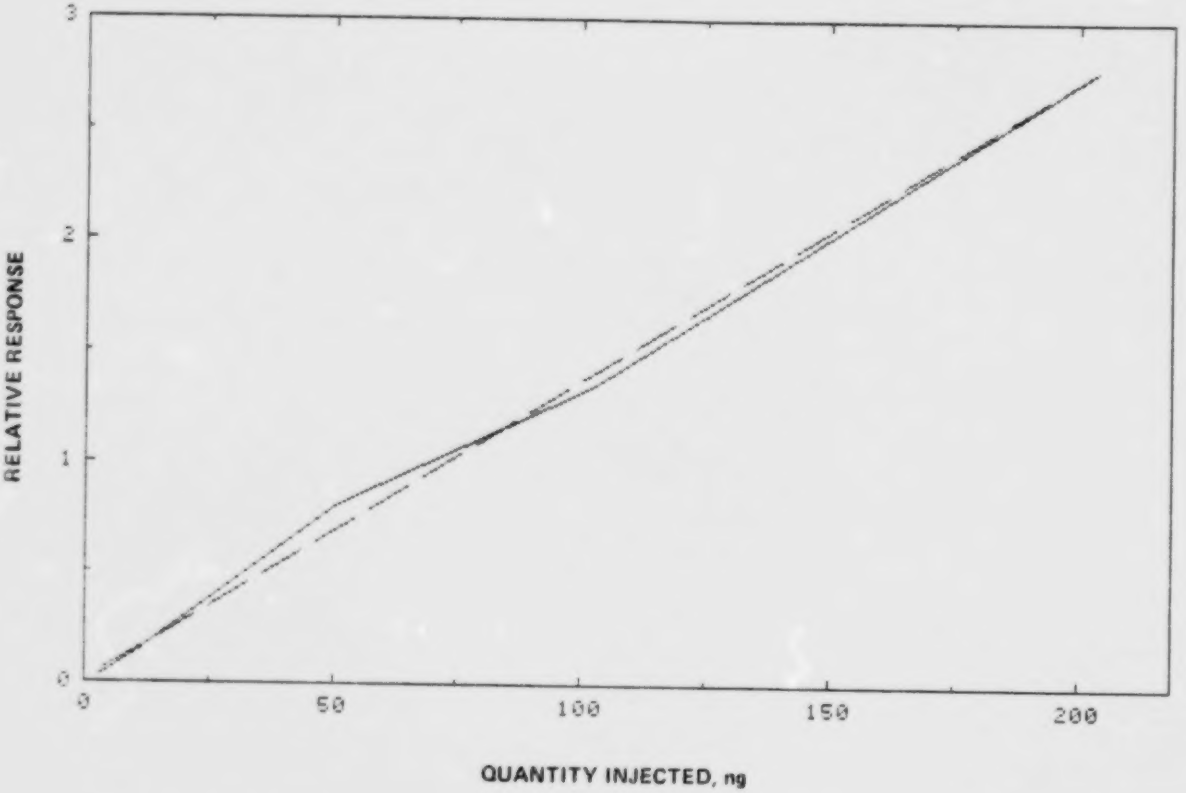


Figure A-8. Calibration curve for the determination of 6,9-dichloro-2-methoxyacridine by GC/FID.

Range: 5.1 - 200 ng
Correlation coefficient: 0.999
Slope: 1.4×10^{-2}
y-Intercept: 1.3×10^{-2}

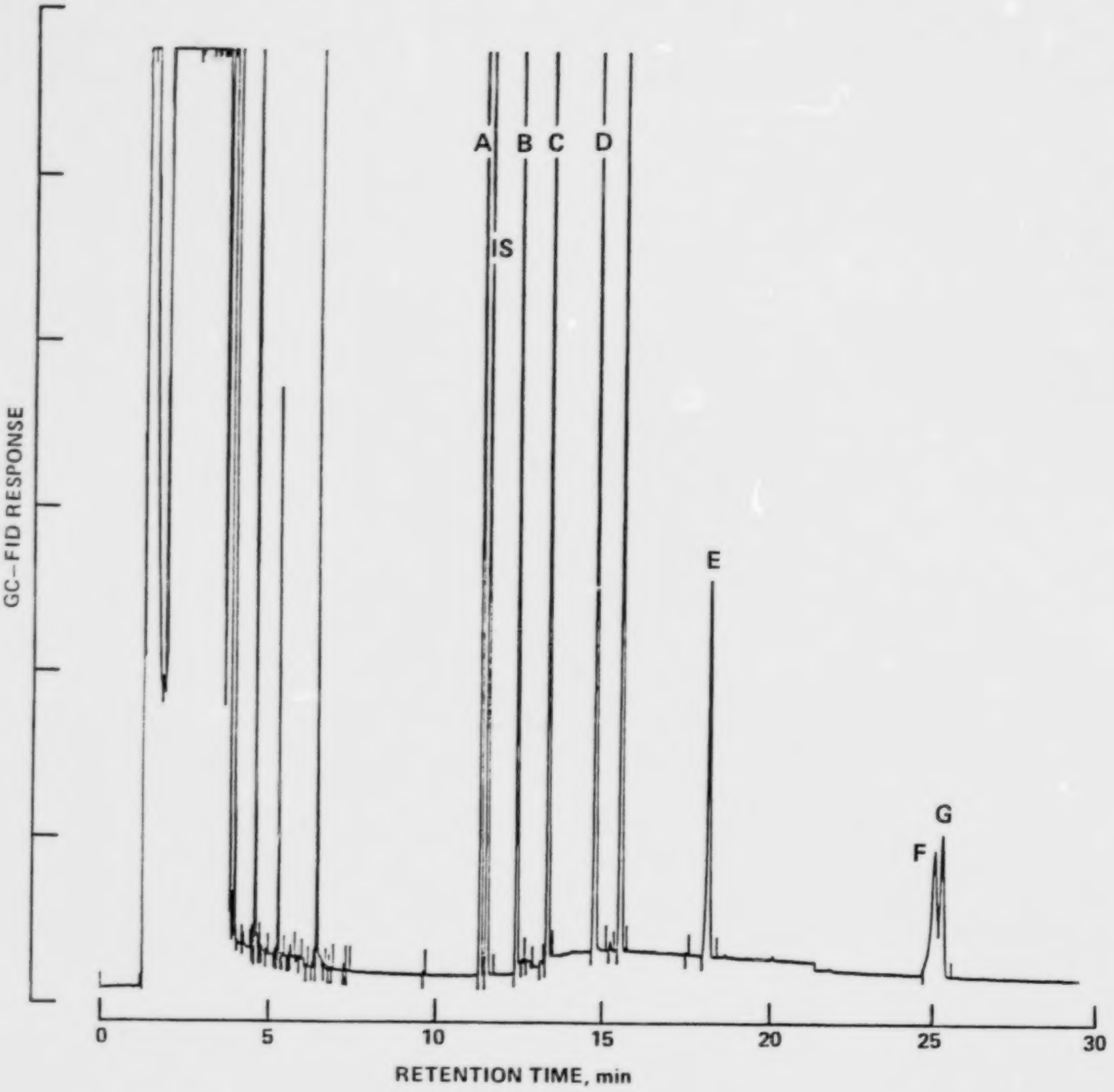


Figure A-9. Chromatogram by GC/FID for

(A) 1-chlorodibenzo-p-dioxin(t_R = 11.5 min),
(B) 2,7-dichlorodibenzo-p-dioxin(t_R = 12.6 min),
(C) 1,2,4-trichlorodibenzo-p-dioxin(t_R = 13.5 min),
(D) 1,2,3,4-tetrachlorodibenzo-p-dioxin(t_R = 14.9 min),
(E) 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin(t_R = 18.4 min),
(F) 1,2,3,4,6,7,8,9-octachlorodibenzo-p-dioxin(t_R = 25.1 min), and
(G) 1,2,3,4,6,7,8,9-octachlorodibenzofuran(t_R = 25.4 min).

Temperature program:
90 °C, 3 min isothermal
90 to 300 °C at 20 °C/min
300 °C, 10 min isothermal

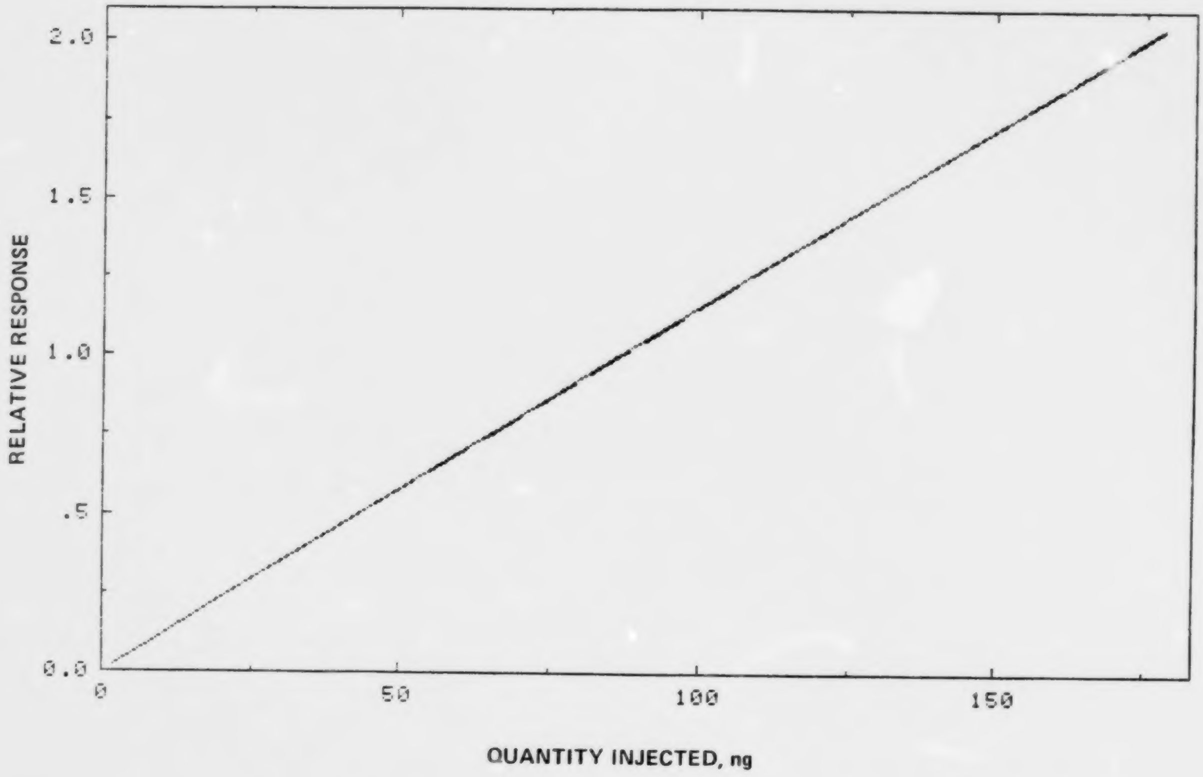


Figure A-10. Calibration curve for the determination of 1-chlorodibenzo-p-dioxin by GC/FID.

Range: 2 - 180 ng
Correlation coefficient: 0.999
Slope: 1.1×10^{-2}
y-Intercept: 1.8×10^{-3}

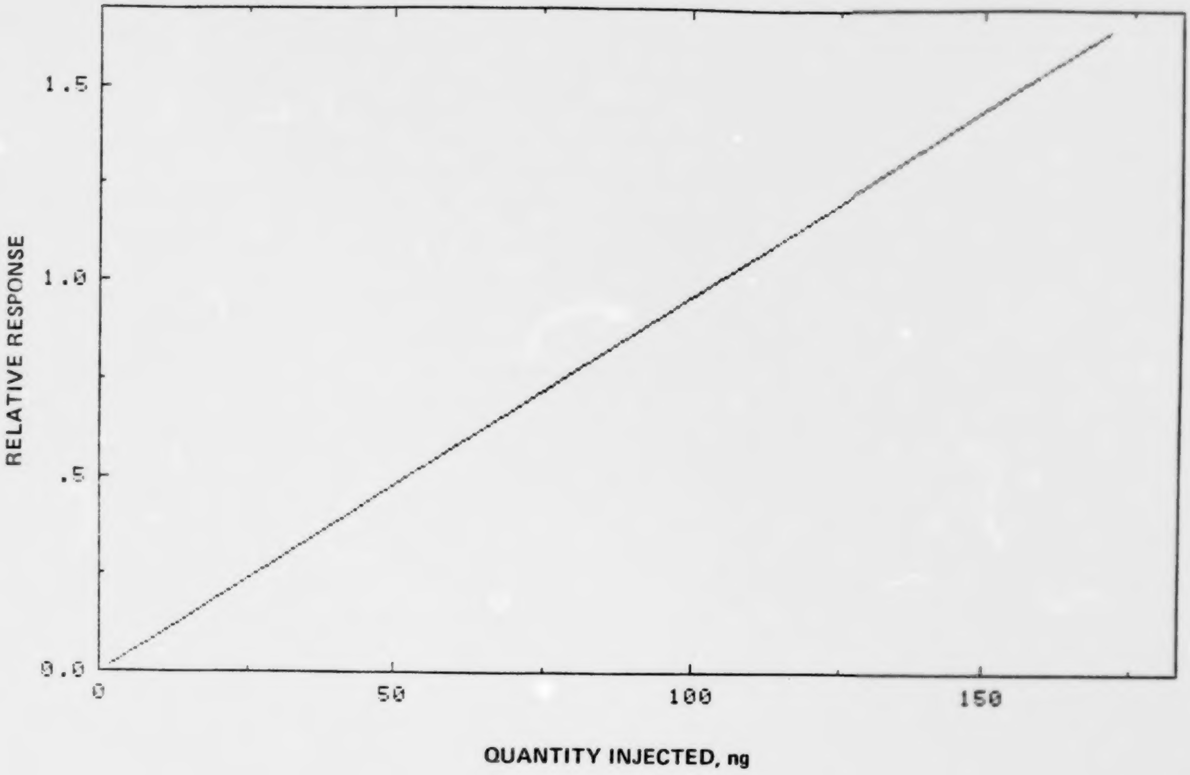


Figure A-11. Calibration curve for the determination of 2,7-dichlorodibenzo-*p*-dioxin by GC/FID.

Range: 2 - 170 ng
Correlation coefficient: 0.999
Slope: 9.7×10^{-3}
y-Intercept: -2.4×10^{-3}

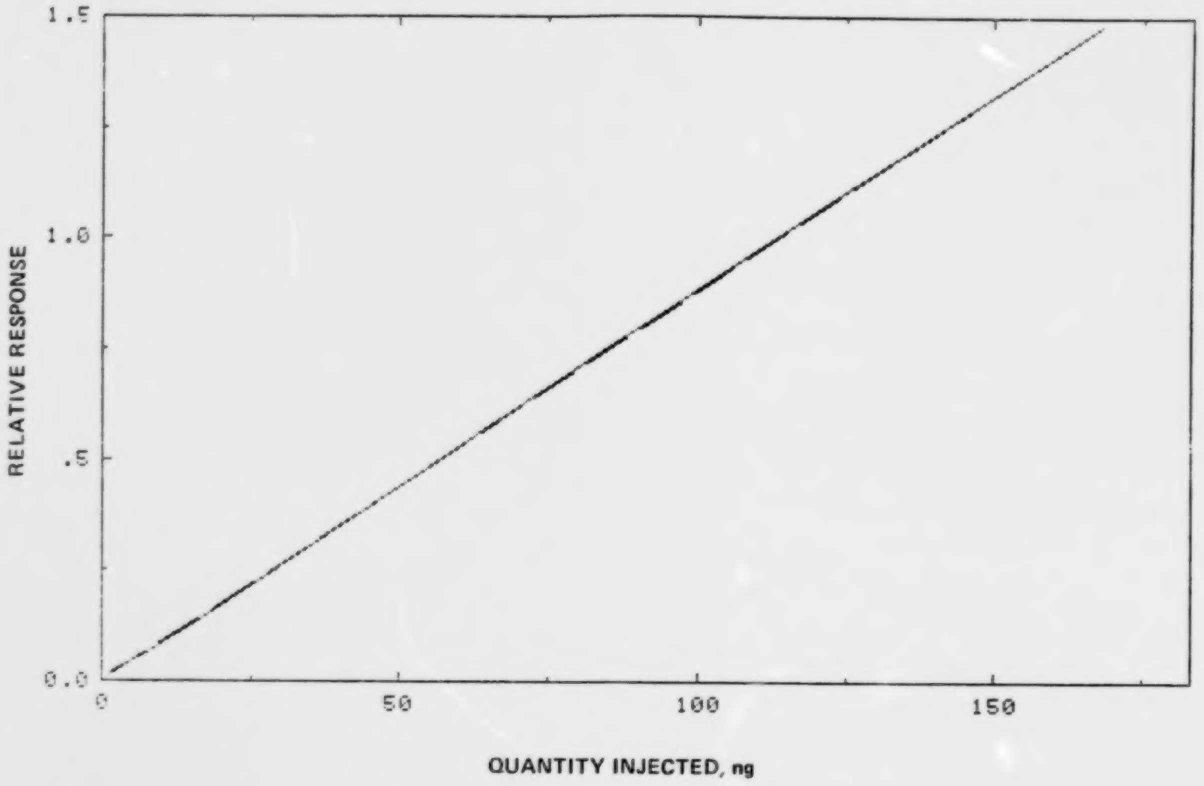


Figure A-12. Calibration curve for the determination of 1,2,4-trichlorodibenzo-p-dioxin by GC/FID.

Range: 5 - 170 ng
Correlation coefficient: 0.999
Slope: 8.8 x 10^{-3}
y-Intercept: -1.6 x 10^{-3}

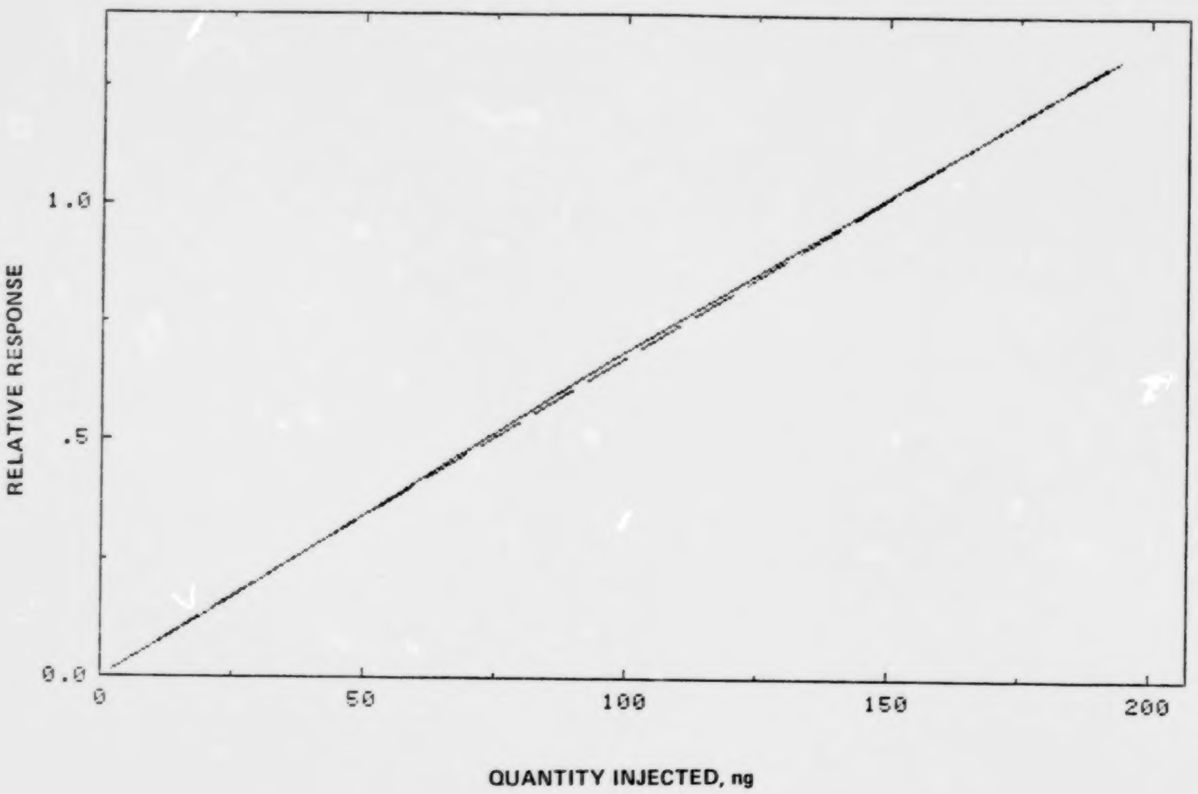


Figure A-13. Calibration curve for the determination of 1,2,3,4-tetrachlorodibenzo-*p*-dioxin by GC/FID.

Range: 2 - 200 ng
Correlation coefficient: 0.999
Slope: 6.8×10^{-3}
y-Intercept: 4.2×10^{-4}

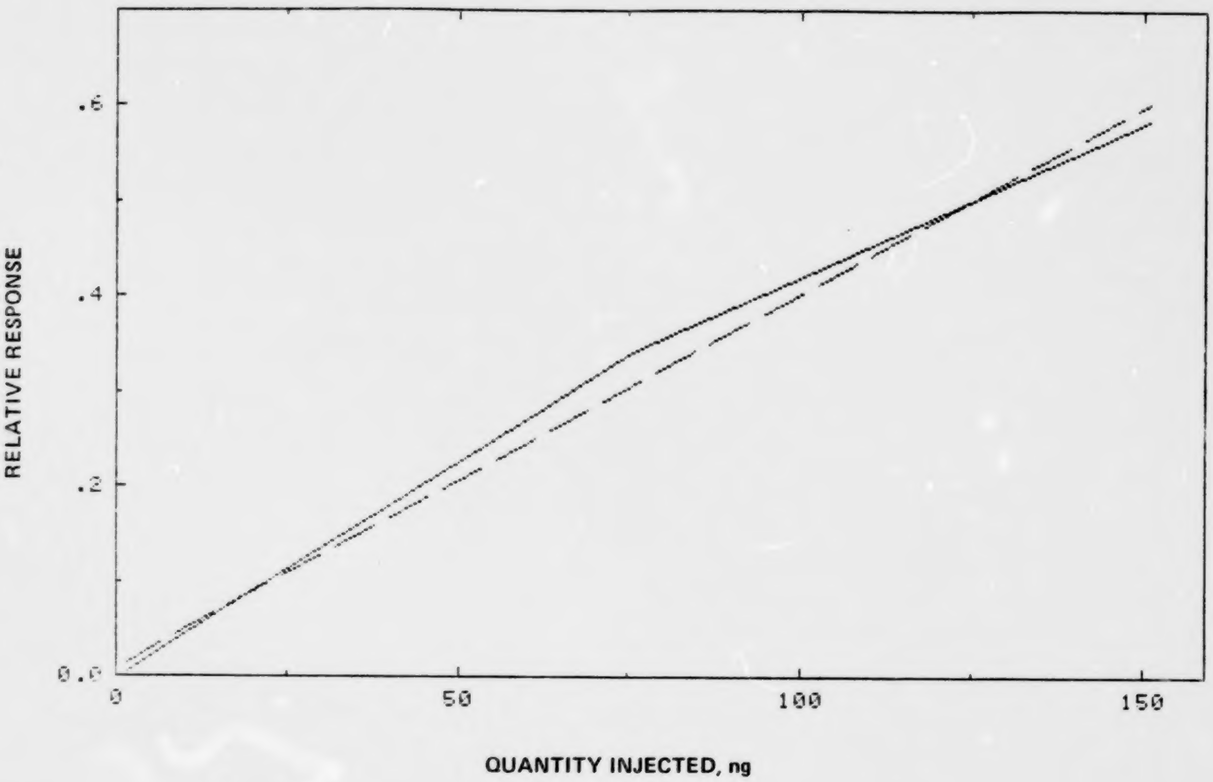


Figure A-14. Calibration curve for the determination of 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin by GC/FID.

Range: 8 - 150 ng
Correlation coefficient: 0.997
Slope: 3.9×10^{-3}
y-Intercept: 8.9×10^{-3}

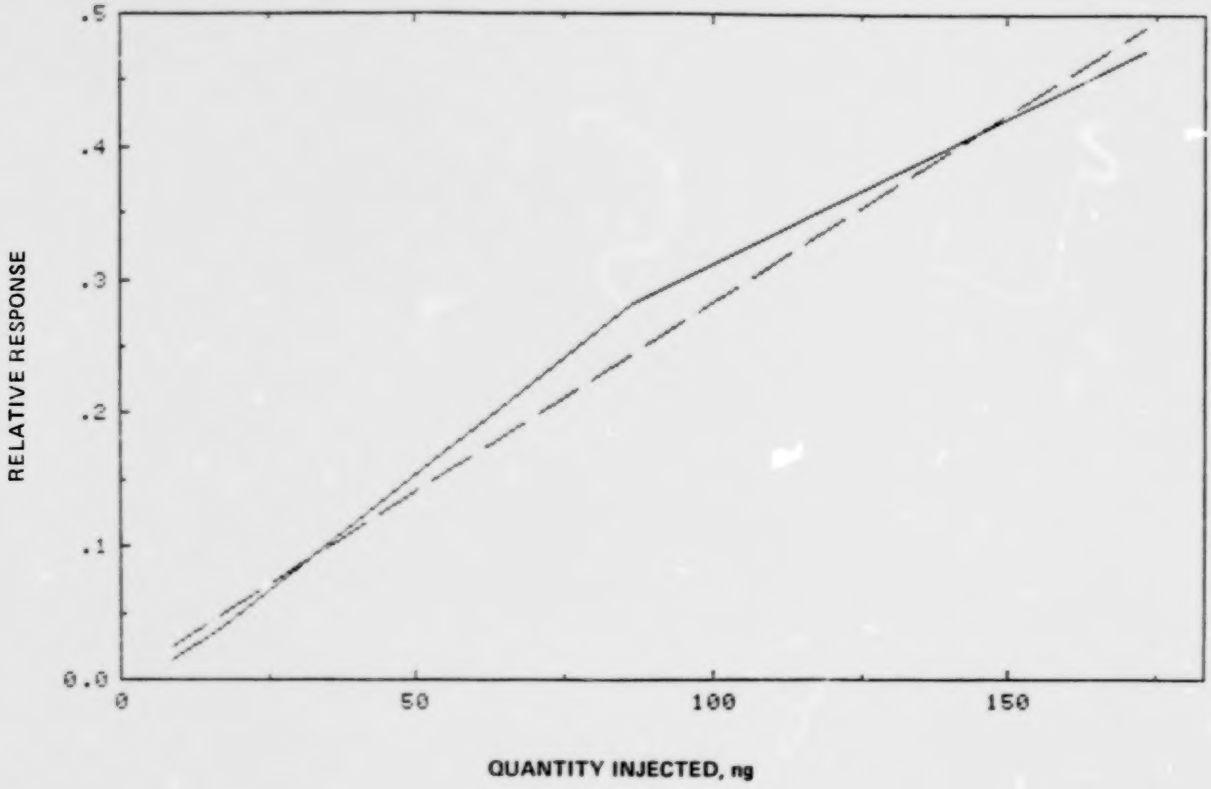


Figure A-15. Calibration curve for the determination of 1,2,3,4,6,7,8,9-octachlorodibenzo-p-dioxin by GC/FID.

Range: 20 - 170 ng
Correlation coefficient: 0.993
Slope: 2.8×10^{-3}
y-Intercept: 6.1×10^{-4}

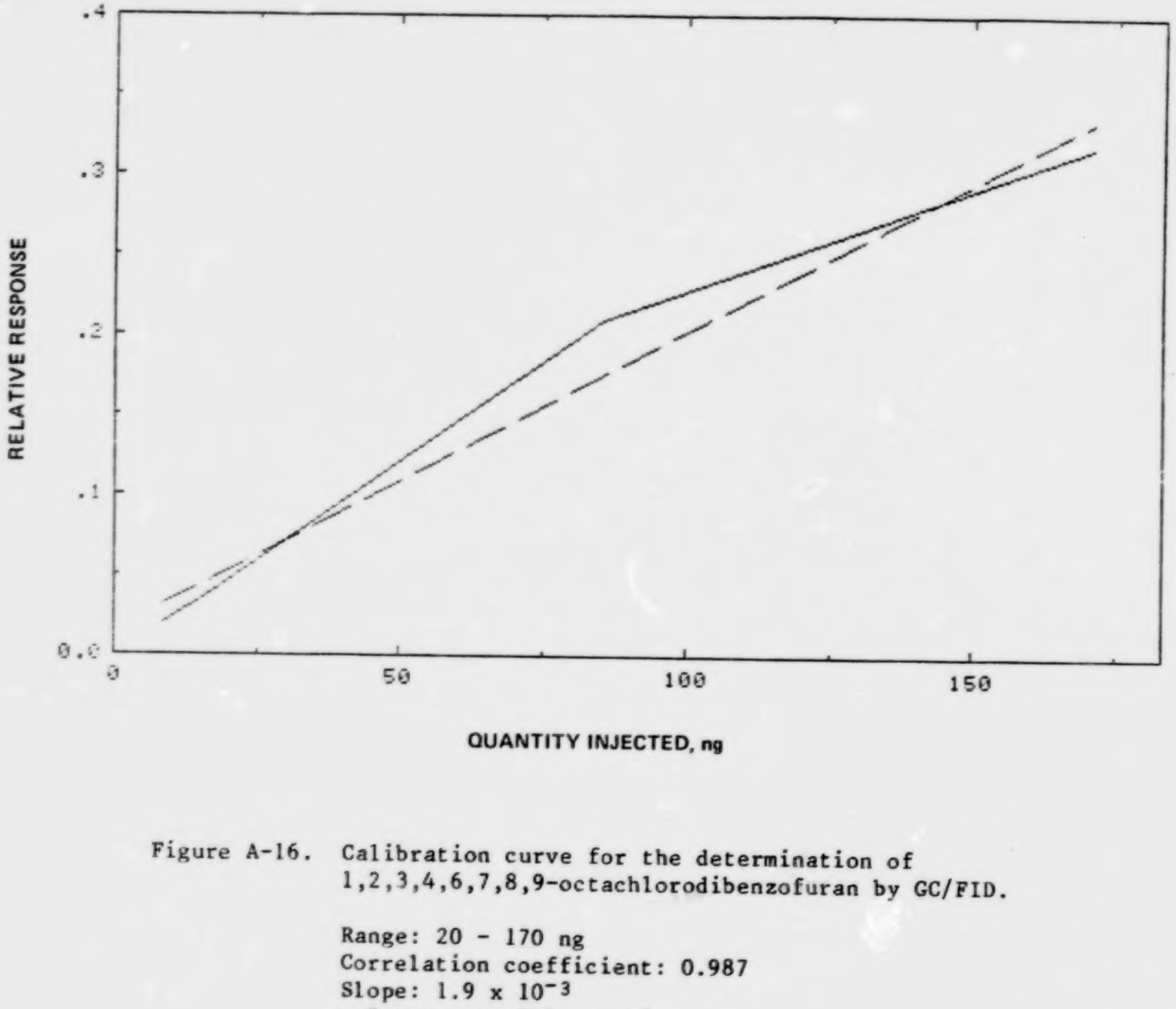


Figure A-16. Calibration curve for the determination of 1,2,3,4,6,7,8,9-octachlorodibenzofuran by GC/FID.

Range: 20 - 170 ng
Correlation coefficient: 0.987
Slope: 1.9 x 10^{-3}
y-Intercept: 1.5 x 10^{-2}

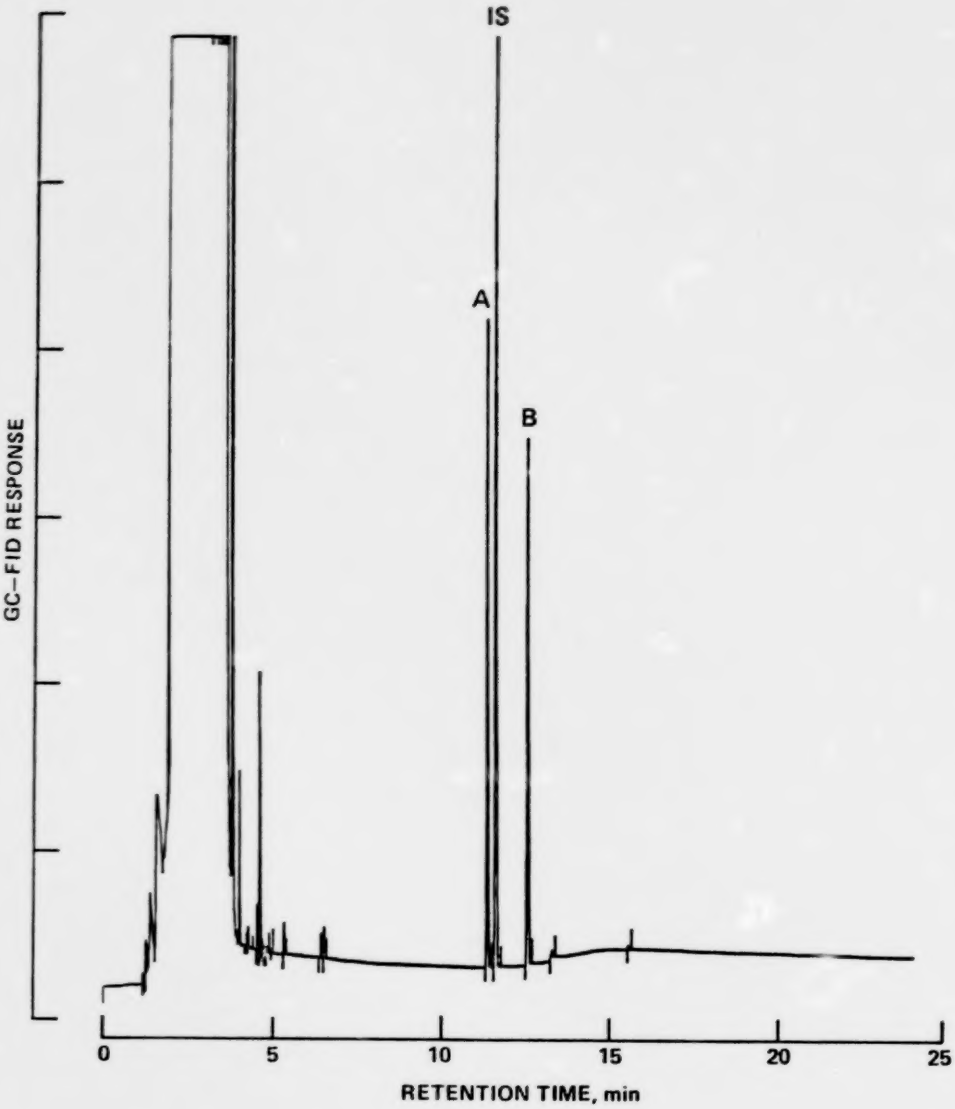


Figure A-17. Chromatogram by GC/FID for

(A) 2-chlorodibenzo-p-dioxin(t_R = 11.4 min) and
(B) 2,3-dichlorodibenzo-p-dioxin(t_R = 12.6 min).

Temperature program:
90 °C, 3 min isothermal
90 to 300 °C at 20 °C/min
300 °C, 10 min isothermal

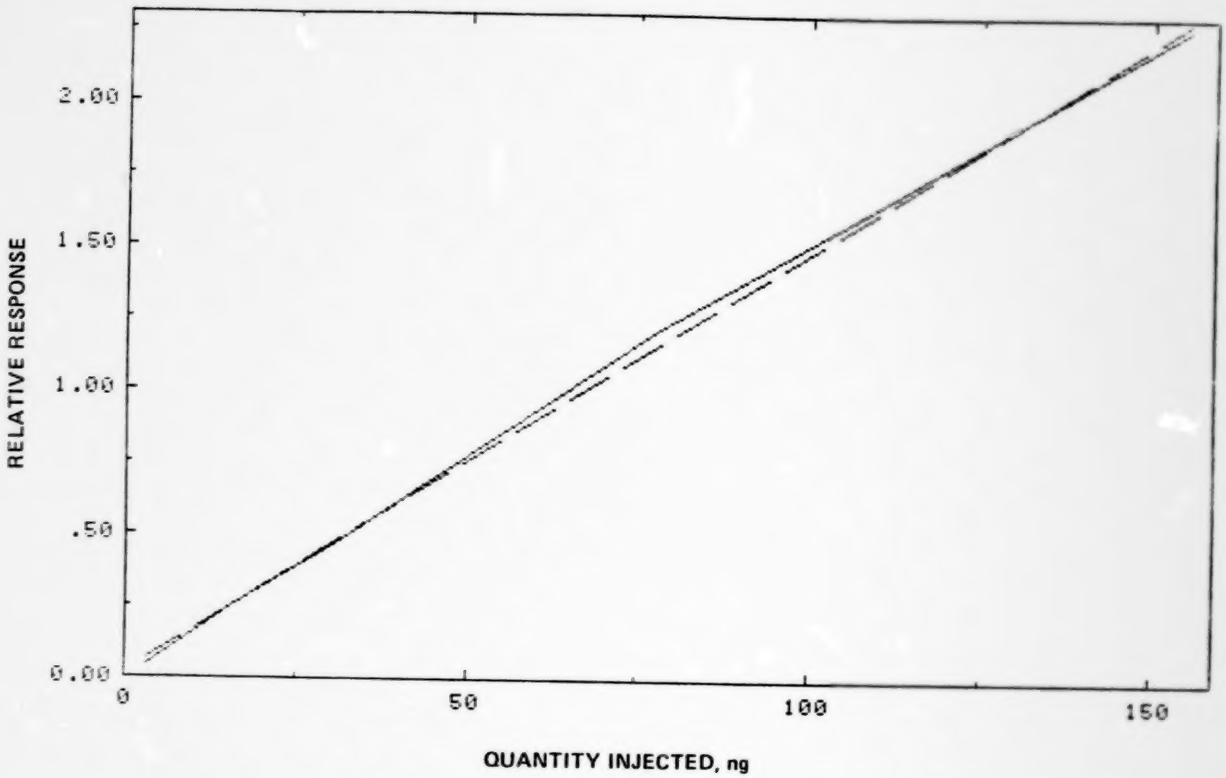


Figure A-18. Calibration curve for the determination of 2-chlorodibenzo-p-dioxin by GC/FID.

Range: 10 - 160 ng
Correlation coefficient: 0.999
Slope: 1.5×10^{-2}
y-Intercept: 2.2×10^{-2}

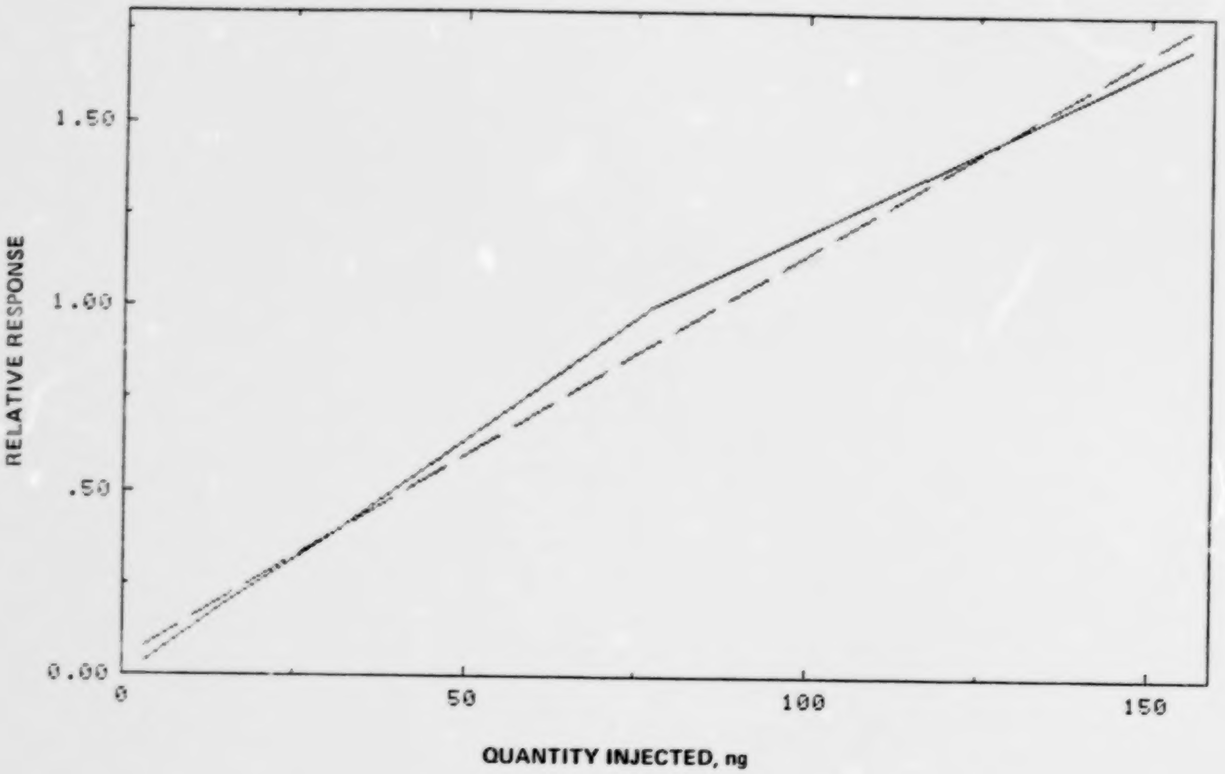


Figure A-19. Calibration curve for the determination of 2,3-dichlorodibenzo-*p*-dioxin by GC/FID.

Range: 10 - 160 ng
Correlation coefficient: 0.996
Slope: 1.1×10^{-2}
y-Intercept: 4.5×10^{-2}

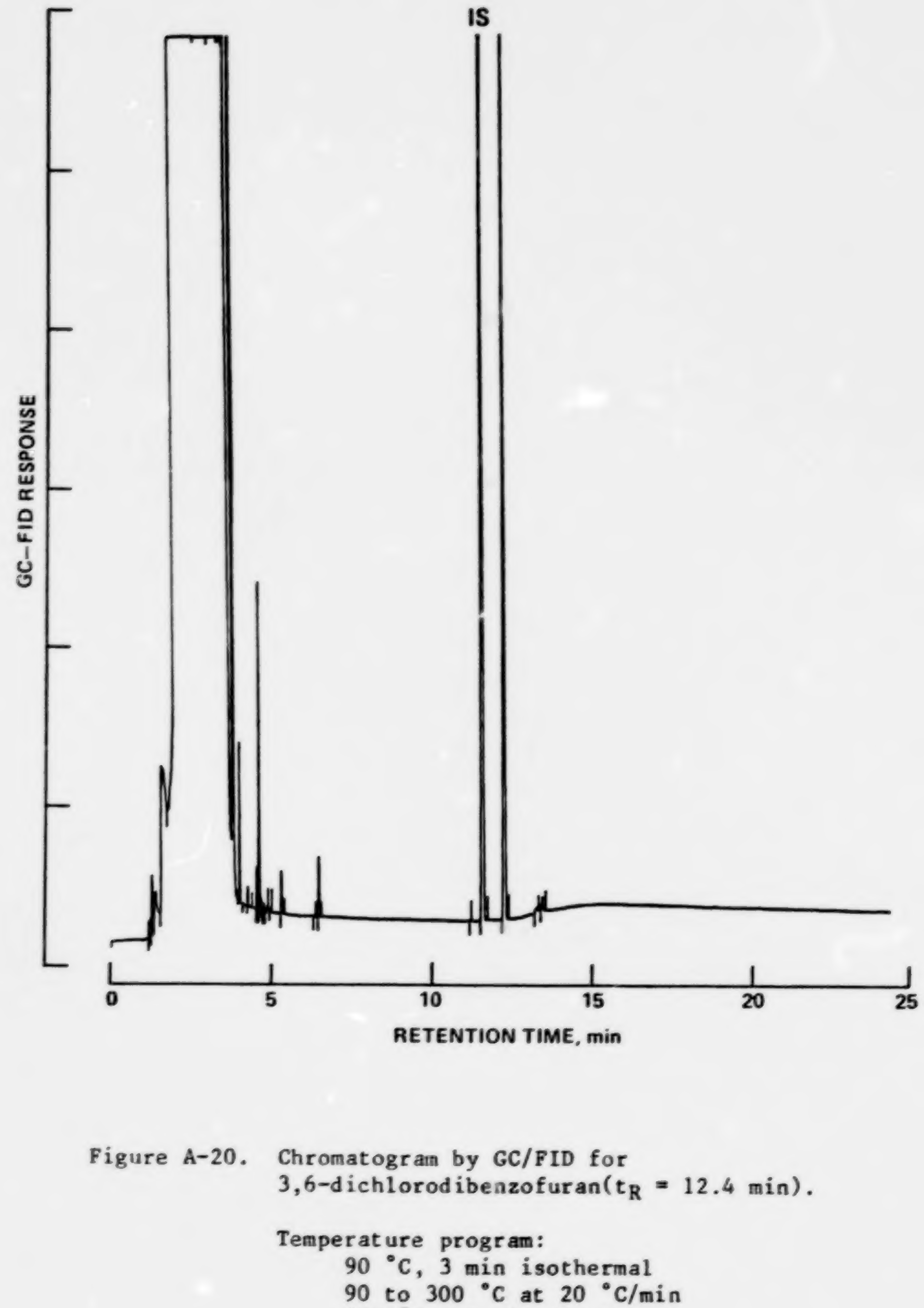


Figure A-20. Chromatogram by GC/FID for 3,6-dichlorodibenzofuran(t_R = 12.4 min).

Temperature program:
90 °C, 3 min isothermal
90 to 300 °C at 20 °C/min
300 °C, 10 min isothermal

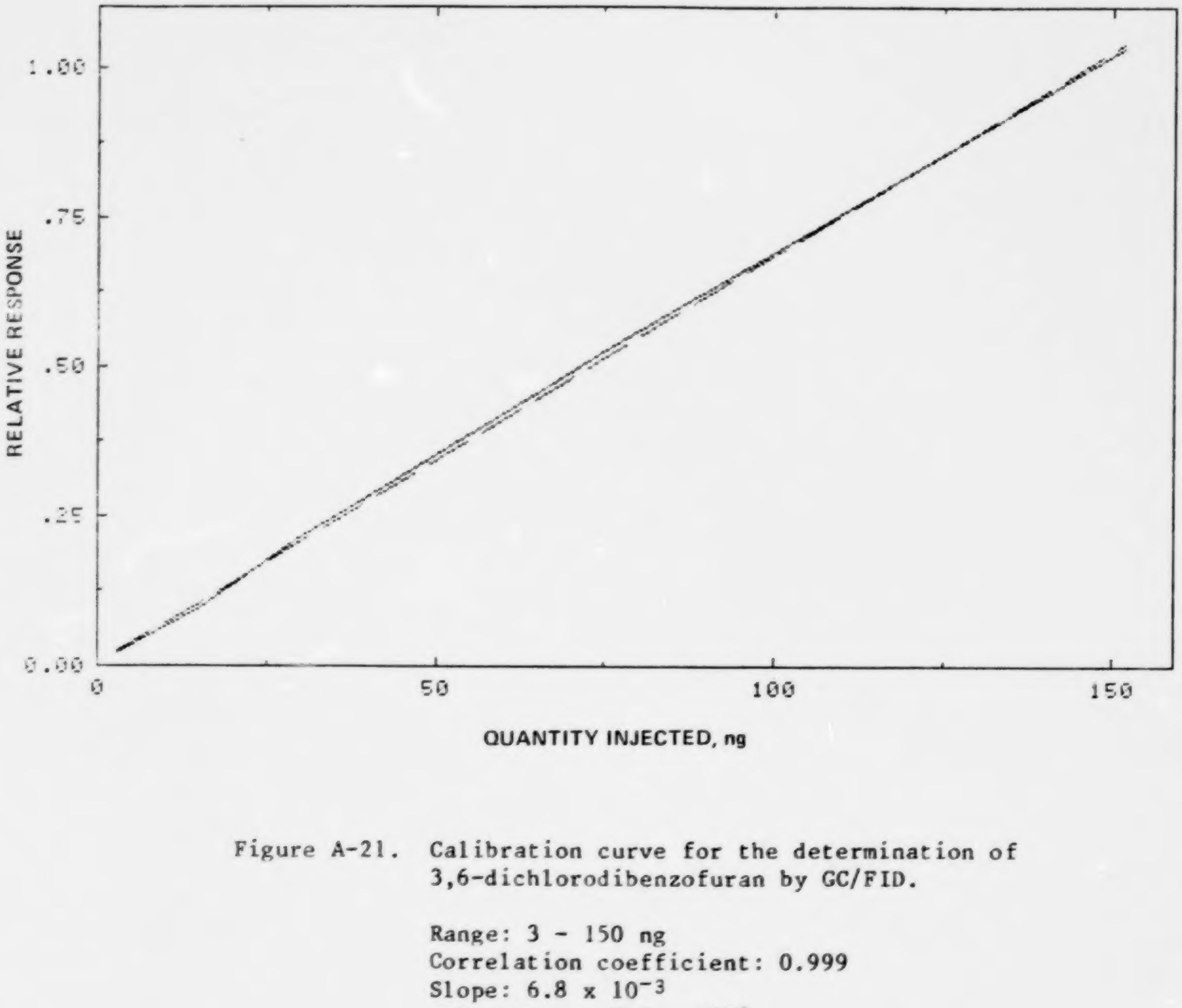


Figure A-21. Calibration curve for the determination of 3,6-dichlorodibenzofuran by GC/FID.

Range: 3 - 150 ng
Correlation coefficient: 0.999
Slope: 6.8×10^{-3}
y-Intercept: 3.0×10^{-3}

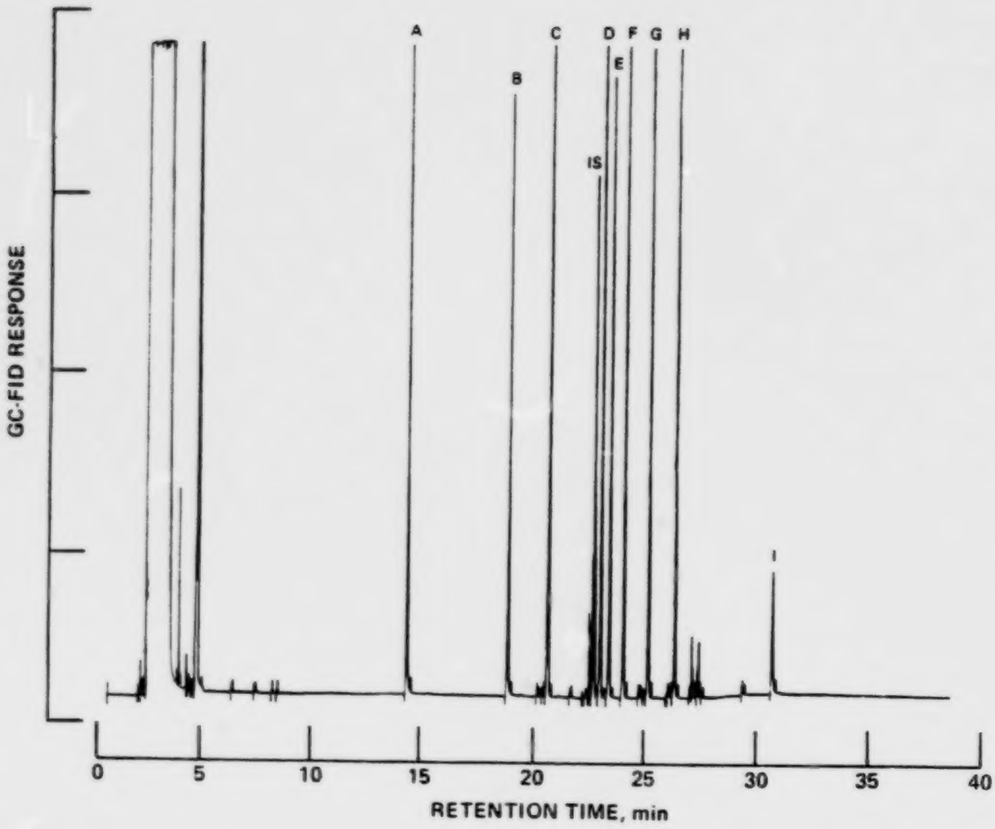


Figure A-22. Chromatogram by GC/FID for

(A) 1-fluoronaphthalene(t_R = 14.0 min),
(B) 3-bromoquinoline(t_R = 18.6 min),
(C) 1-bromo-4-methylnaphthalene(t_R = 20.5 min),
(D) 5-bromoacenaphthene(t_R = 22.5 min),
(E) 1-chloro-8-nitronaphthalene(t_R = 23.3 min),
(F) 2-bromofluorene(t_R = 23.9 min),
(G) 9-chloroanthracene(t_R = 25.1 min),
(H) 9-bromoanthracene(t_R = 26.4 min), and
(I) 3,6-dibromocarbazole(t_R = 30.8 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

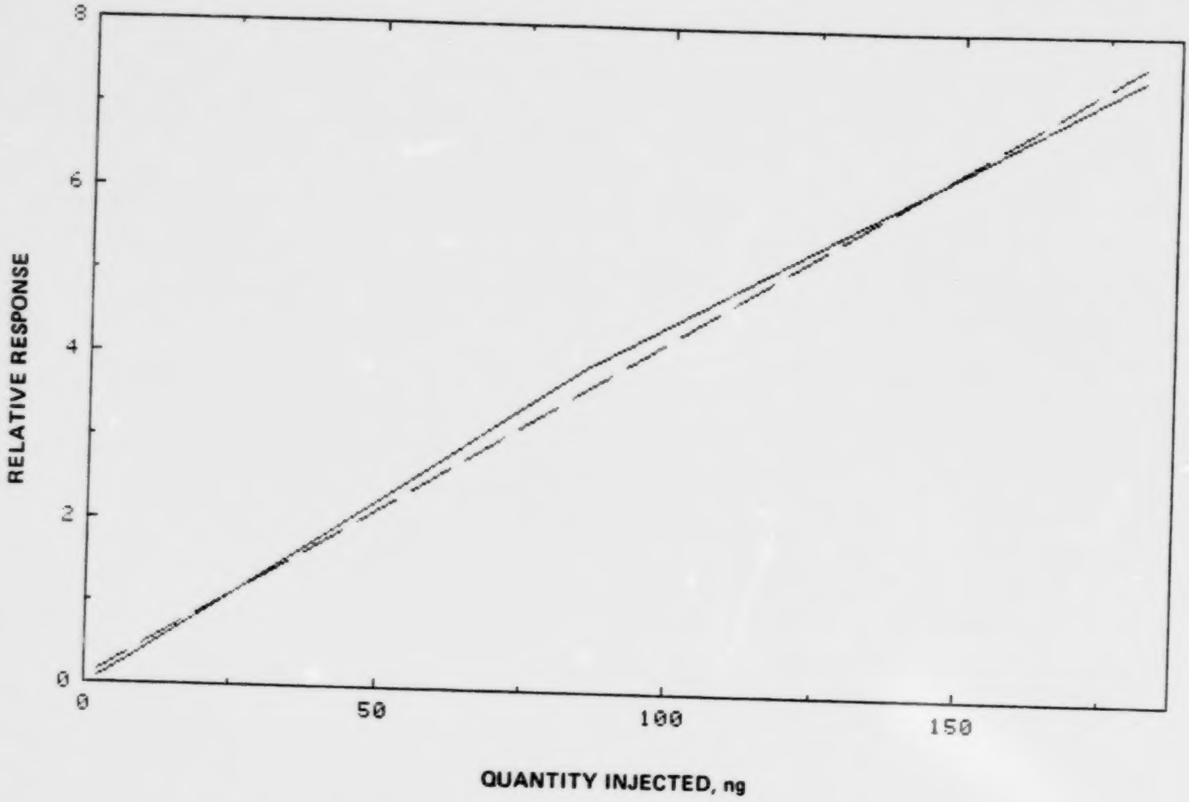


Figure A-23. Calibration curve for the determination of 1-fluoronaphthalene by GC/FID.

Range: 4.3 - 180 ng
Correlation coefficient: 0.999
Slope: 4.2×10^{-2}
y-Intercept: 6.2×10^{-2}

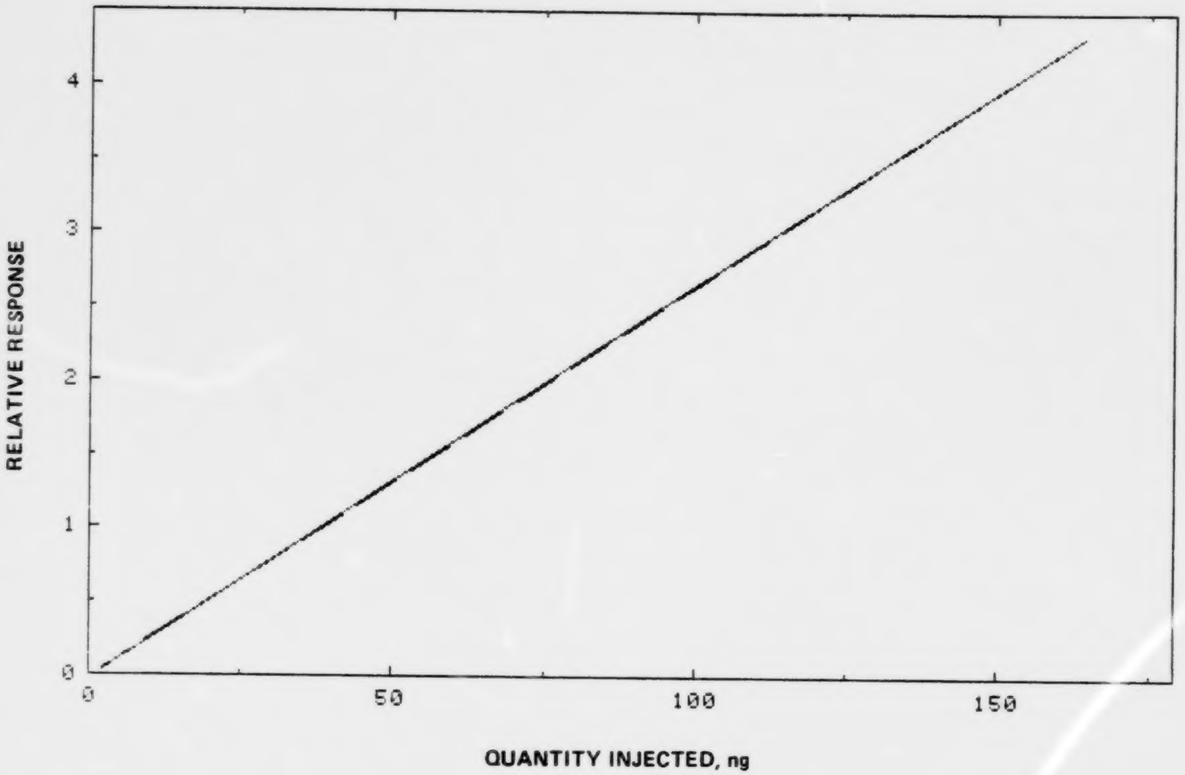


Figure A-24. Calibration curve for the determination of 3-bromoquinoline by GC/FID.

Range: 2.0 - 160 ng
Correlation coefficient: 0.999
Slope: 2.7×10^{-2}
y-Intercept: -1.8×10^{-2}

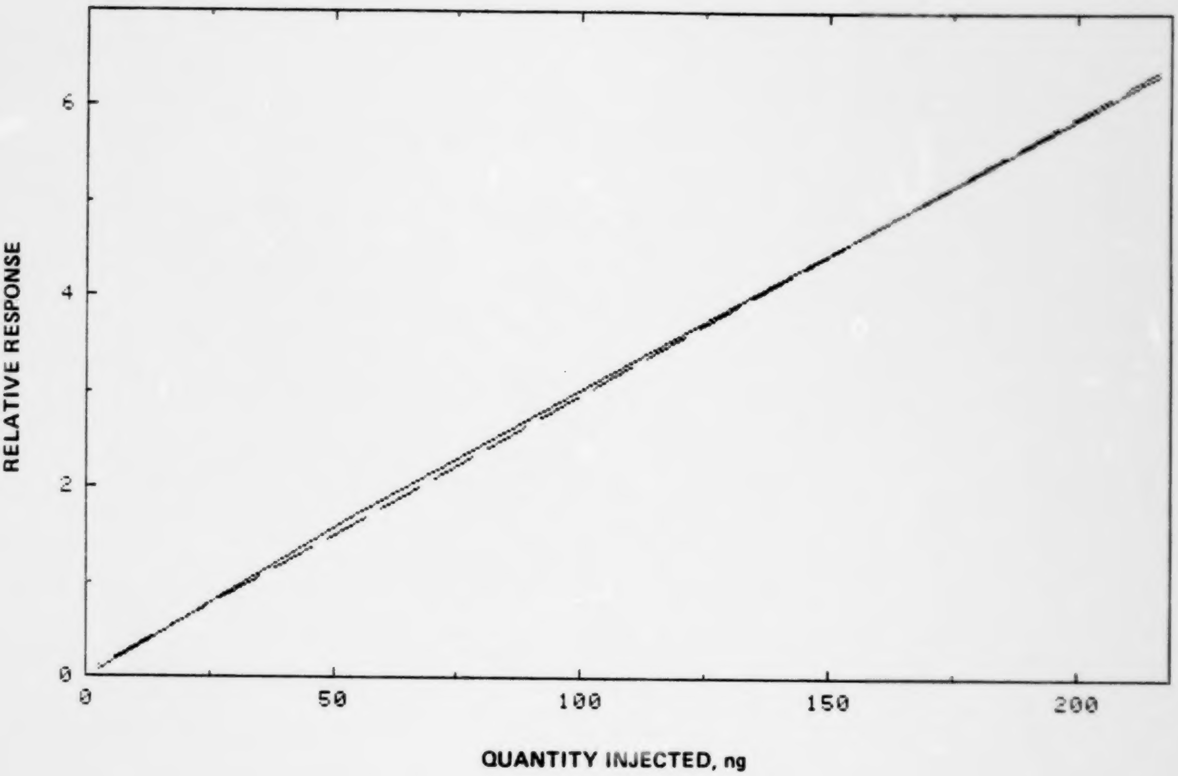


Figure A-25. Calibration curve for the determination of 1-bromo-4-methylnaphthalene by GC/FID.

Range: 2.7 - 220 ng
Correlation coefficient: 0.999
Slope: 2.9×10^{-2}
y-Intercept: 3.1×10^{-2}

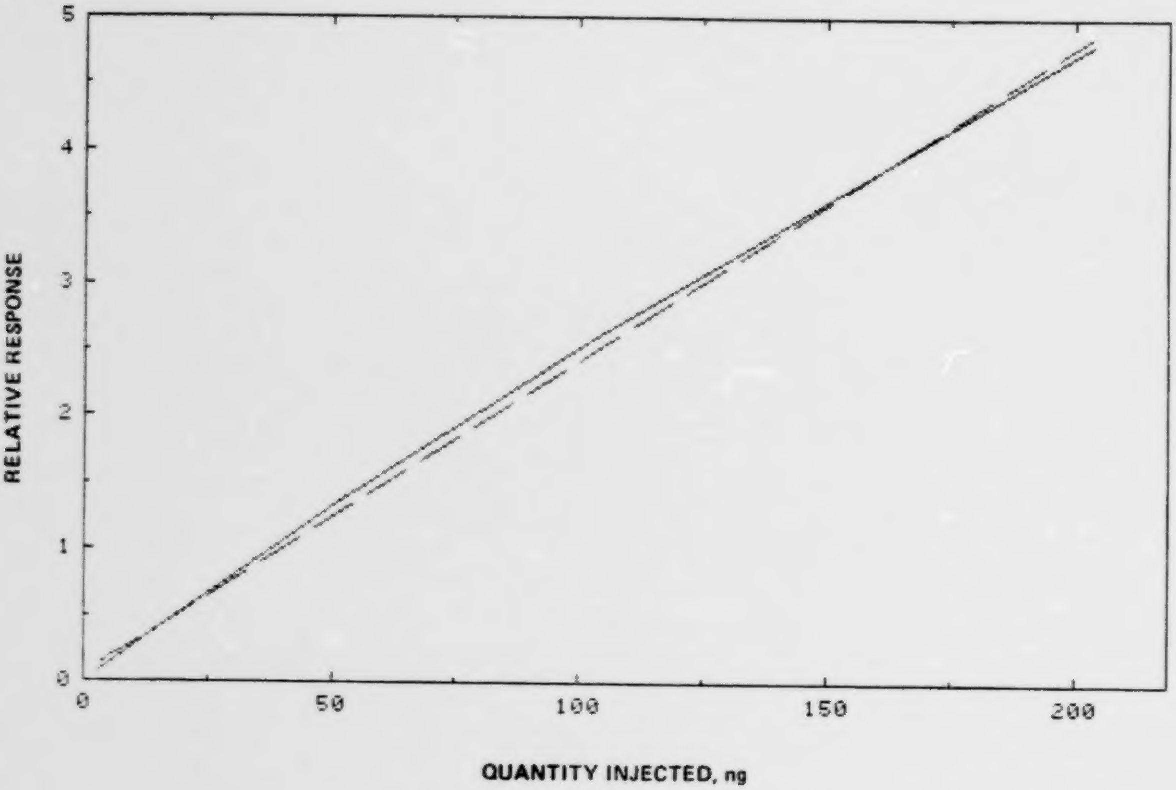


Figure A-26. Calibration curve for the determination of 5-bromoacenaphthene by GC/FID.

Range: 5.1 - 200 ng
Correlation coefficient: 0.999
Slope: 2.4×10^{-2}
y-Intercept: 5.9×10^{-2}

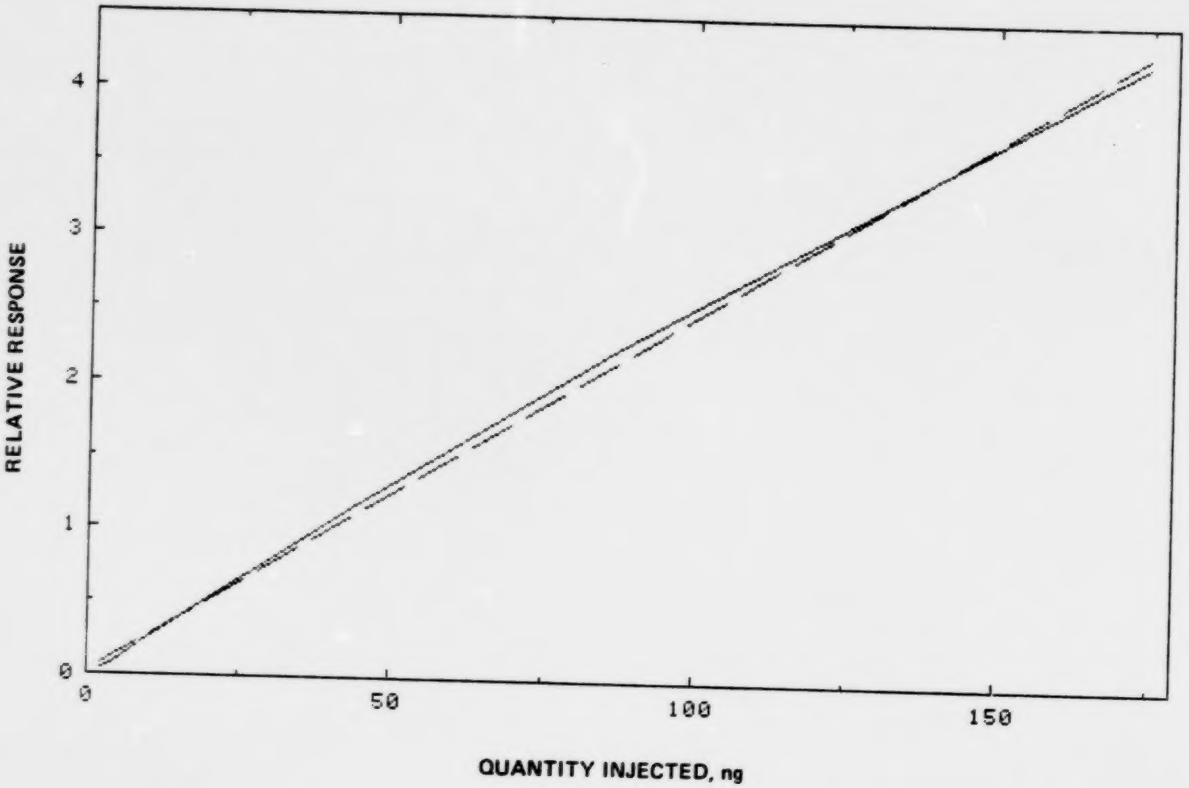


Figure A-27. Calibration curve for the determination of 1-chloro-8-nitronaphthalene by GC/FID.

Range: 4.4 - 170 ng
Correlation coefficient: 0.999
Slope: 2.4×10^{-2}
y-Intercept: 2.6×10^{-2}

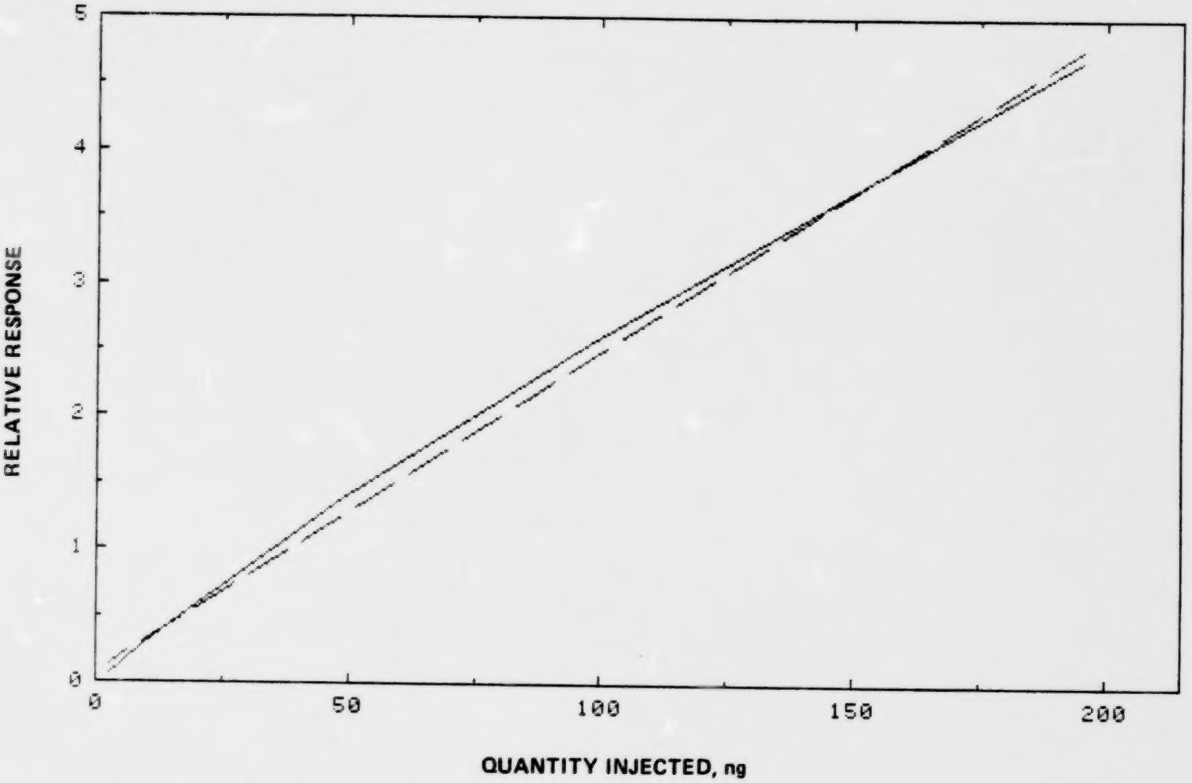


Figure A-28. Calibration curve for the determination of 2-bromofluorene by GC/FID.

Range: 9.8 - 200 ng
Correlation coefficient: 0.997
Slope: 2.3 x 10^{-2}
y-Intercept: 1.1 x 10^{-1}

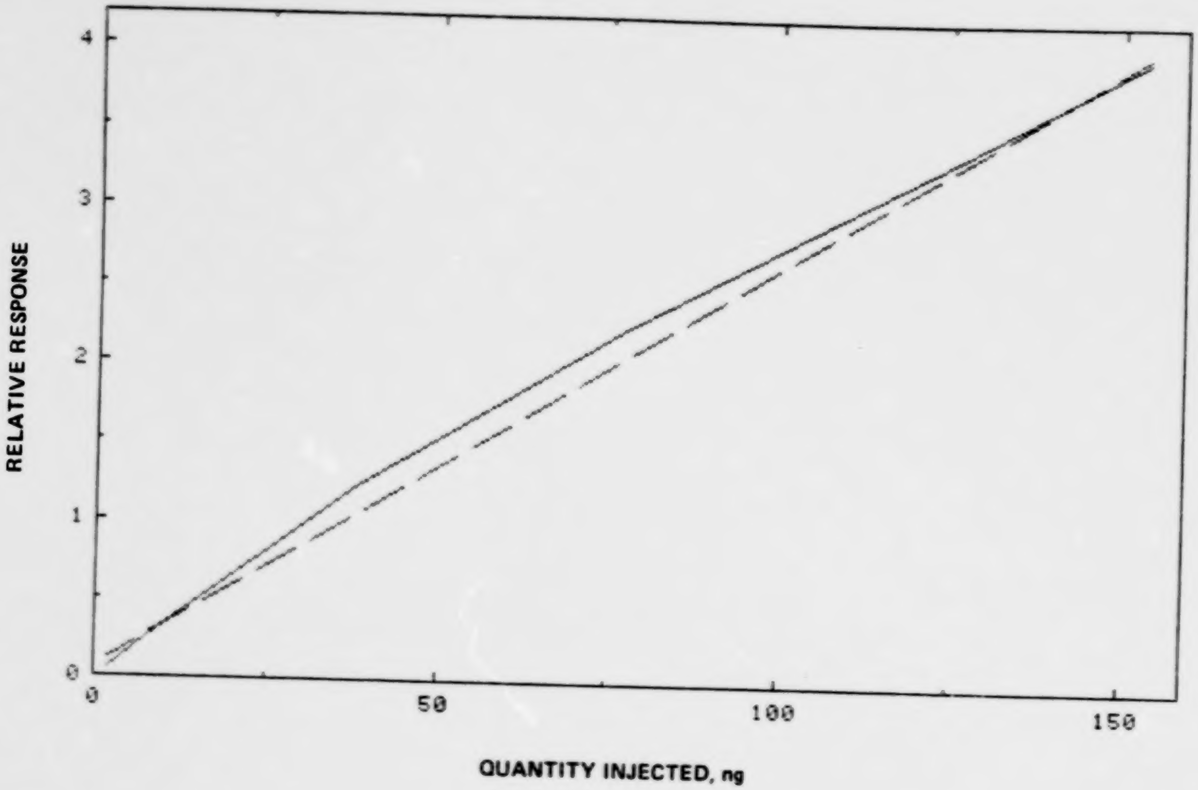


Figure A-29. Calibration curve for the determination of 9-chloroanthracene by GC/FID.

Range: 3.8 - 150 ng
Correlation coefficient: 0.997
Slope: 2.6×10^{-2}
y-Intercept: 9.4×10^{-2}

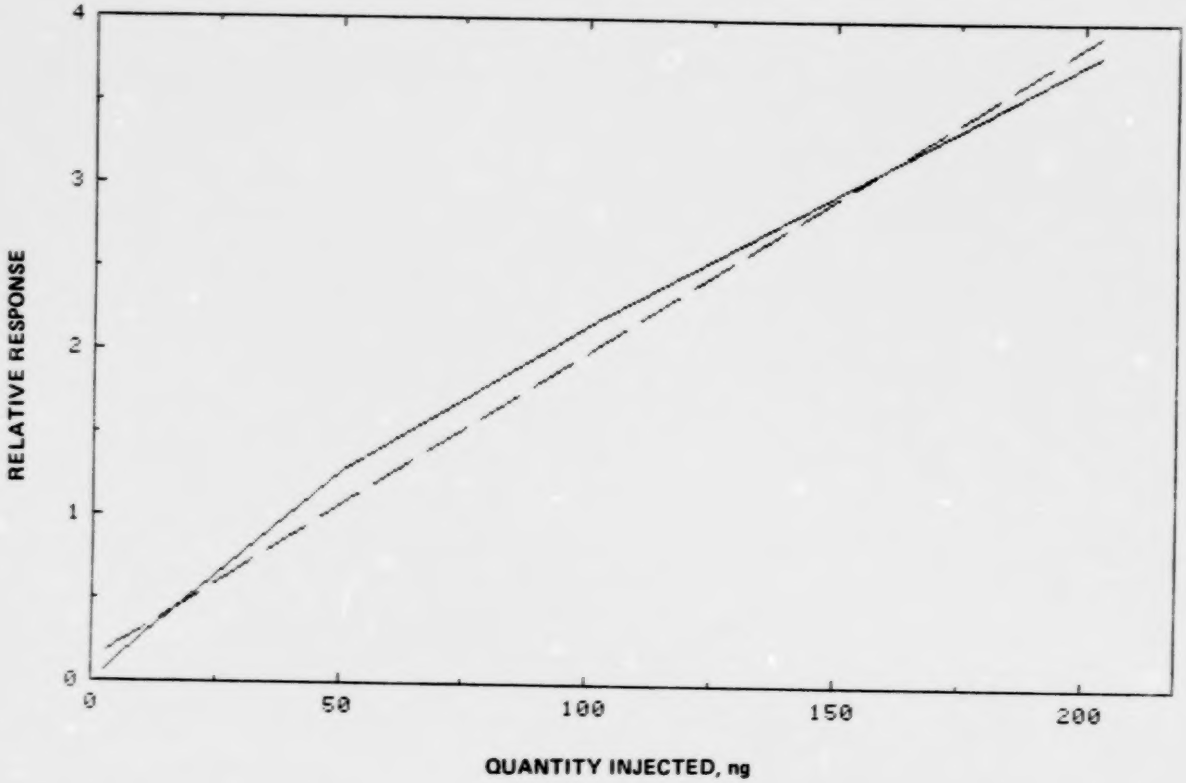


Figure A-30. Calibration curve for the determination of 9-bromoanthracene by GC/FID.

Range: 5.1 - 200 ng
Correlation coefficient: 0.995
Slope: 1.9×10^{-2}
y-Intercept: 1.3×10^{-1}

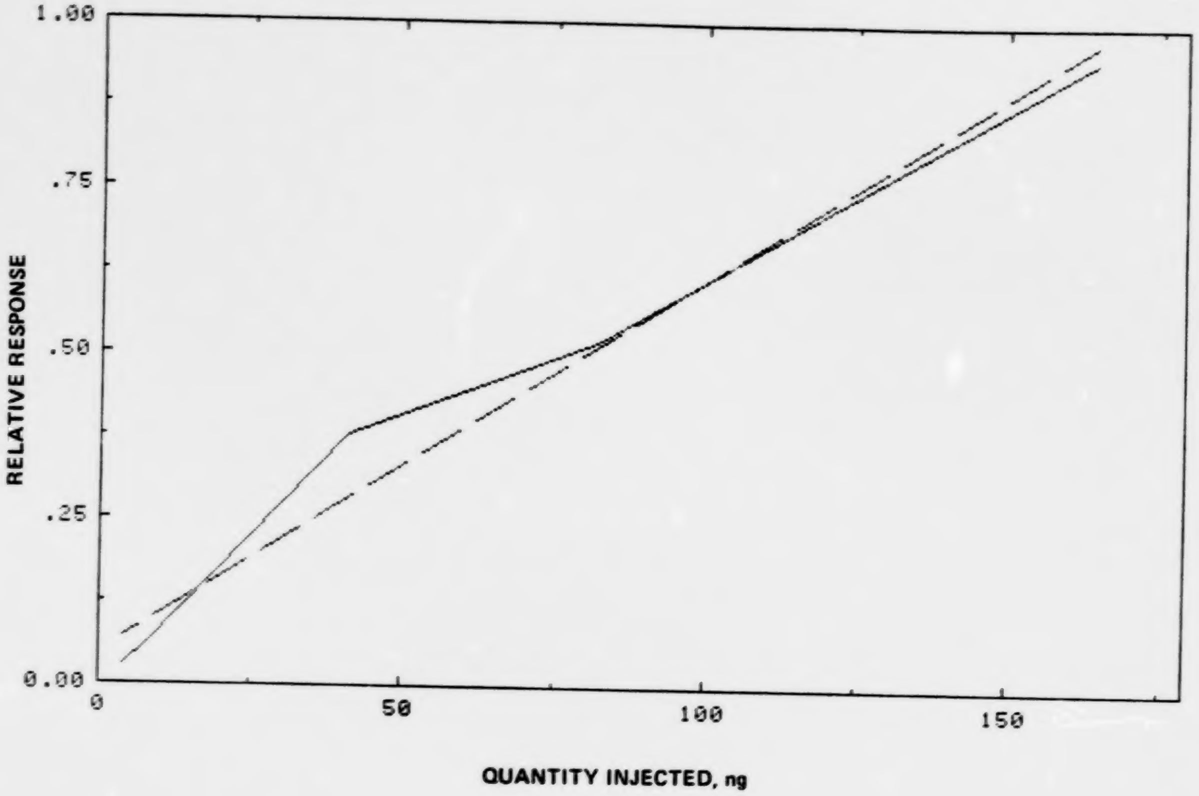


Figure A-31. Calibration curve for the determination of 3,6-dibromocarbazole by GC/FID.

Range: 8.2 - 160 ng
Correlation coefficient: 0.988
Slope: 5.6 x 10^{-3}
y-Intercept: 5.1 x 10^{-2}

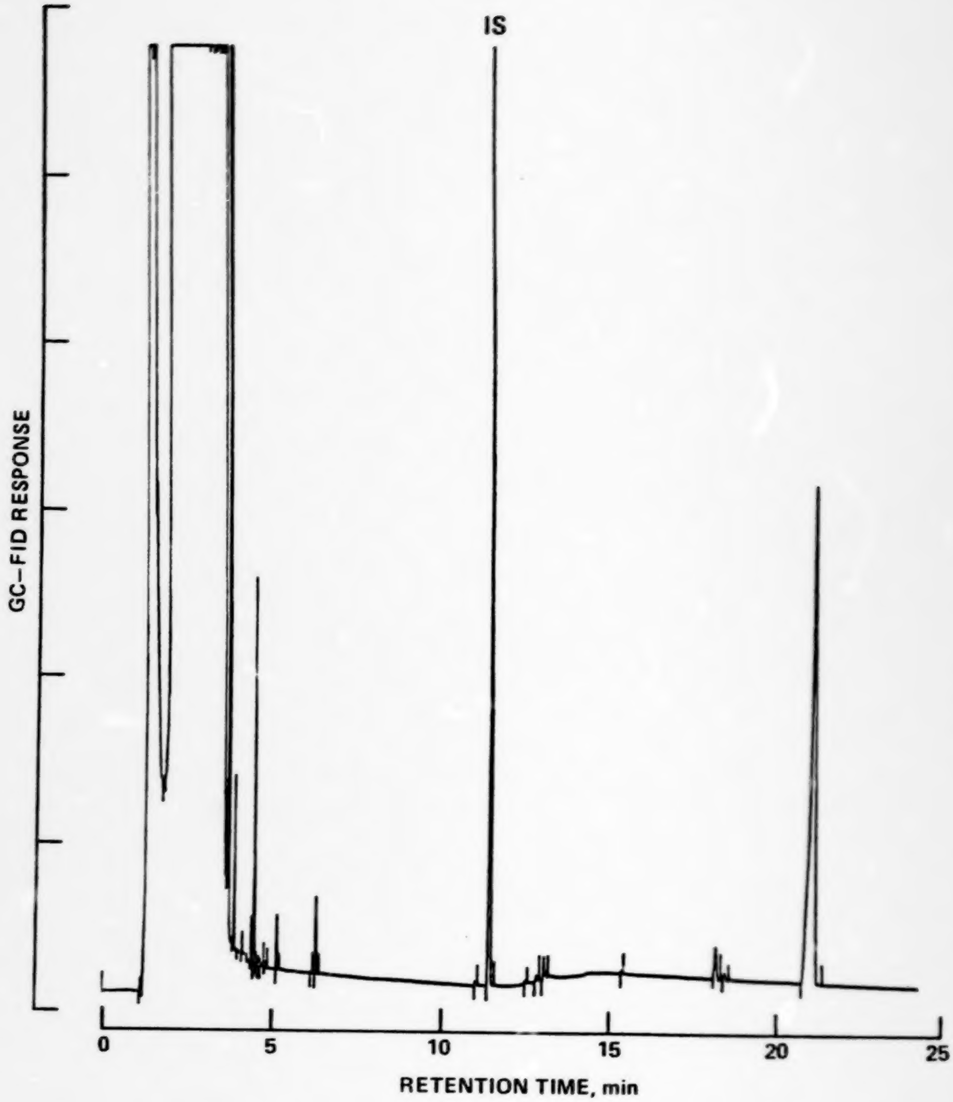


Figure A-32. Chromatogram by GC/FID for 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin(t_R = 21.3 min).

Temperature program:
90 °C, 3 min isothermal
90 to 300 °C at 20 °C/min
300 °C, 10 min isothermal

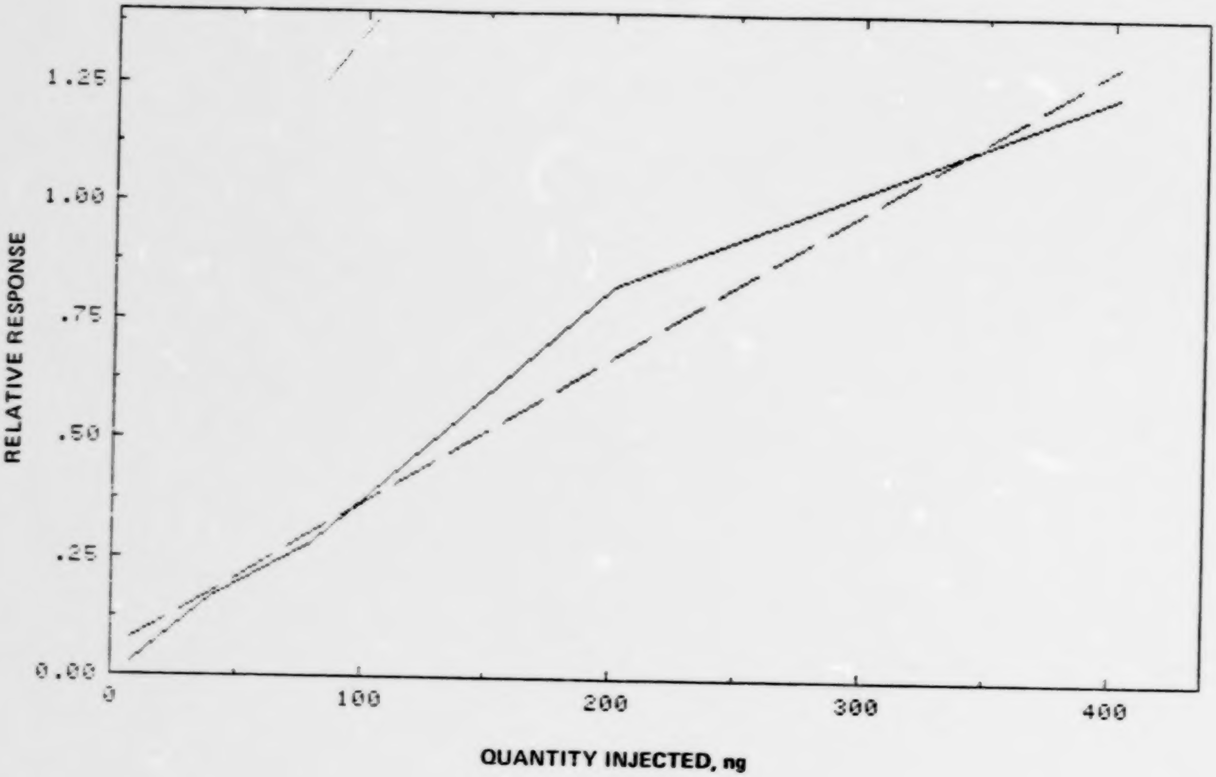


Figure A-33. Calibration curve for the determination of 1,2,3,4,6,7,8-heptachlorodibenzo-*p*-dioxin by GC/FID.

Range: 40 - 400 ng
Correlation coefficient: 0.985
Slope: 3.1×10^{-3}
y-Intercept: 5.2×10^{-2}

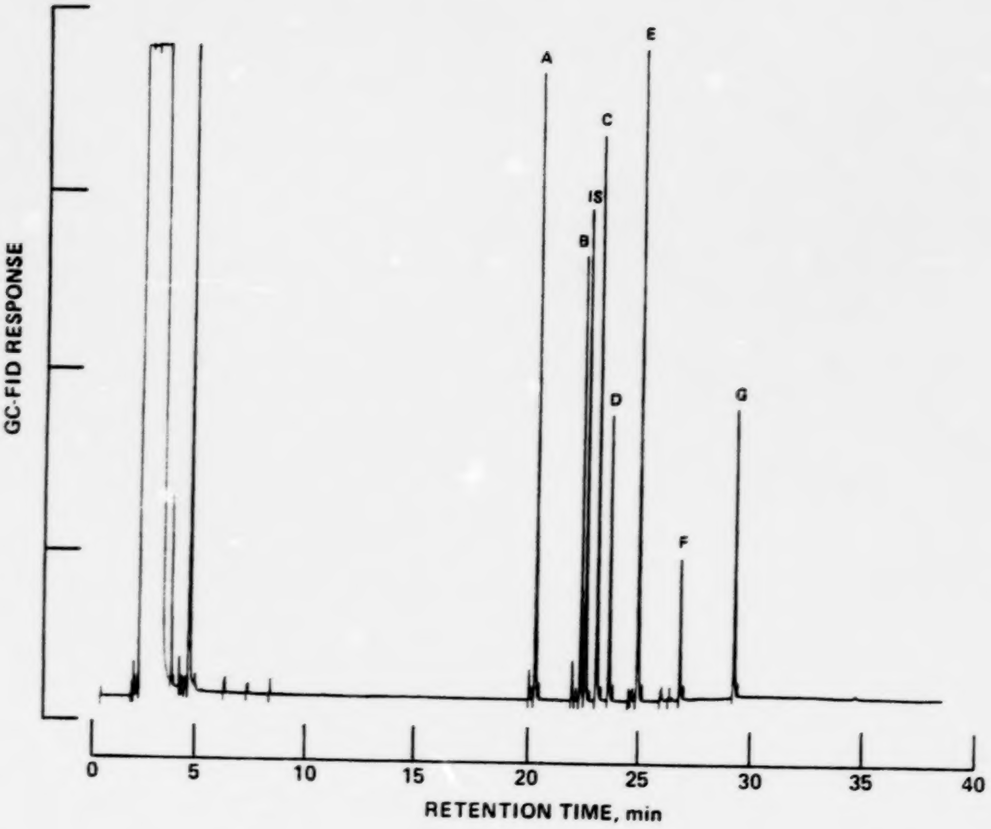


Figure A-34. Chromatogram by GC/FID for

(A) 1-iodonaphthalene(t_R = 20.3 min),
(B) 1-amino-4-chloronaphthalene(t_R = 22.3 min),
(C) 9-bromofluorene(t_R = 23.0 min),
(D) 1-amino-4-bromonaphthalene(t_R = 23.6 min),
(E) 2-chloroanthracene(t_R = 24.9 min),
(F) 9-(chloromethyl)anthracene(t_R = 26.9 min), and
(G) 9,10-dibromoanthracene(t_R = 29.4 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

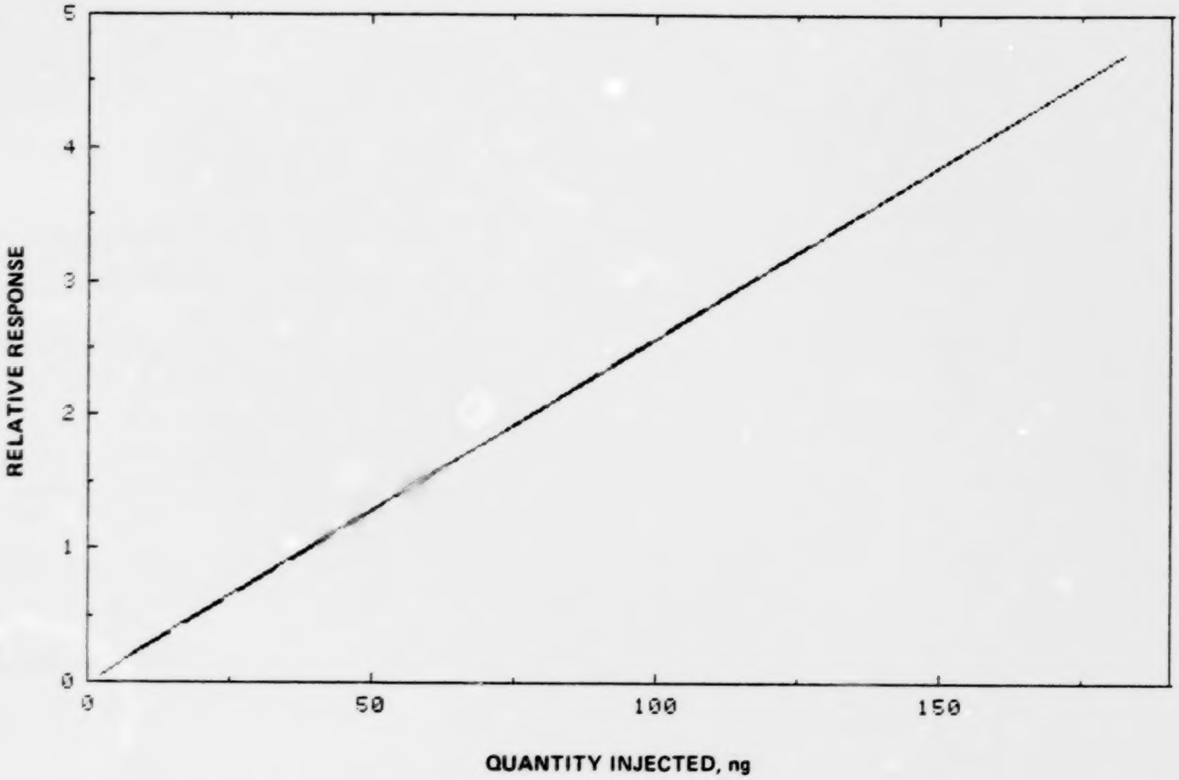


Figure A-35. Calibration curve for the determination of 1-iodonaphthalene by GC/FID.

Range: 2.3 - 180 ng
Correlation coefficient: 0.999
Slope: 2.6×10^{-2}
y-Intercept: -3.4×10^{-3}

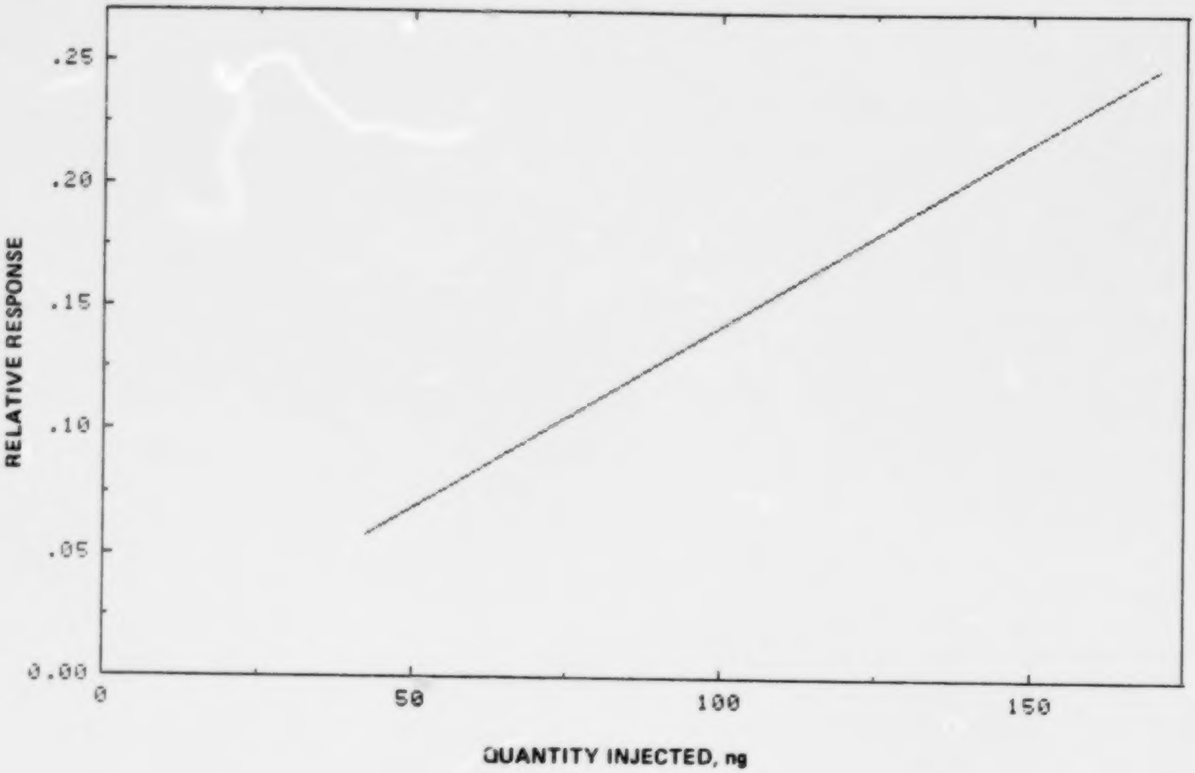


Figure A-36. Calibration curve for the determination of 1-amino-4-chloronaphthalene by GC/FID.

Range: 43 - 170 ng
Correlation coefficient: 0.999
Slope: 1.5×10^{-3}
y-Intercept: -6.3×10^{-3}

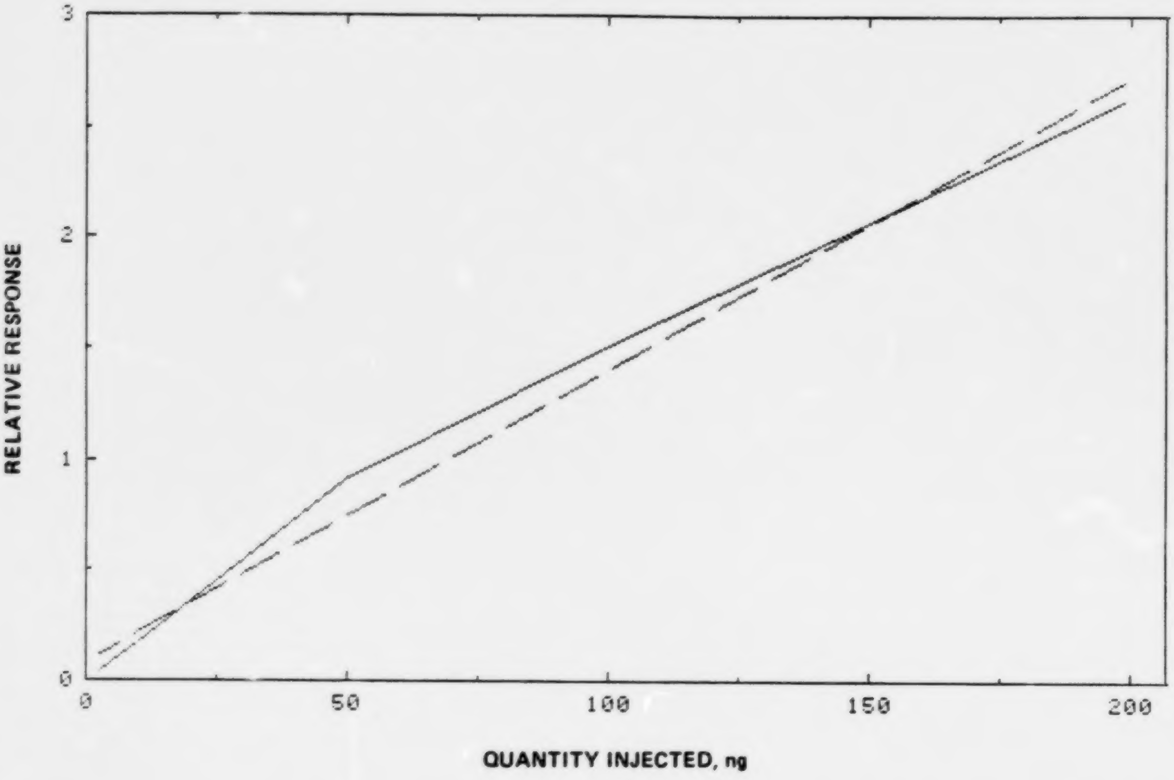


Figure A-37. Calibration curve for the determination of 9-bromofluorene by GC/FID.

Range: 10 - 200 ng
Correlation coefficient: 0.994
Slope: 1.3×10^{-2}
y-Intercept: 8.5×10^{-2}

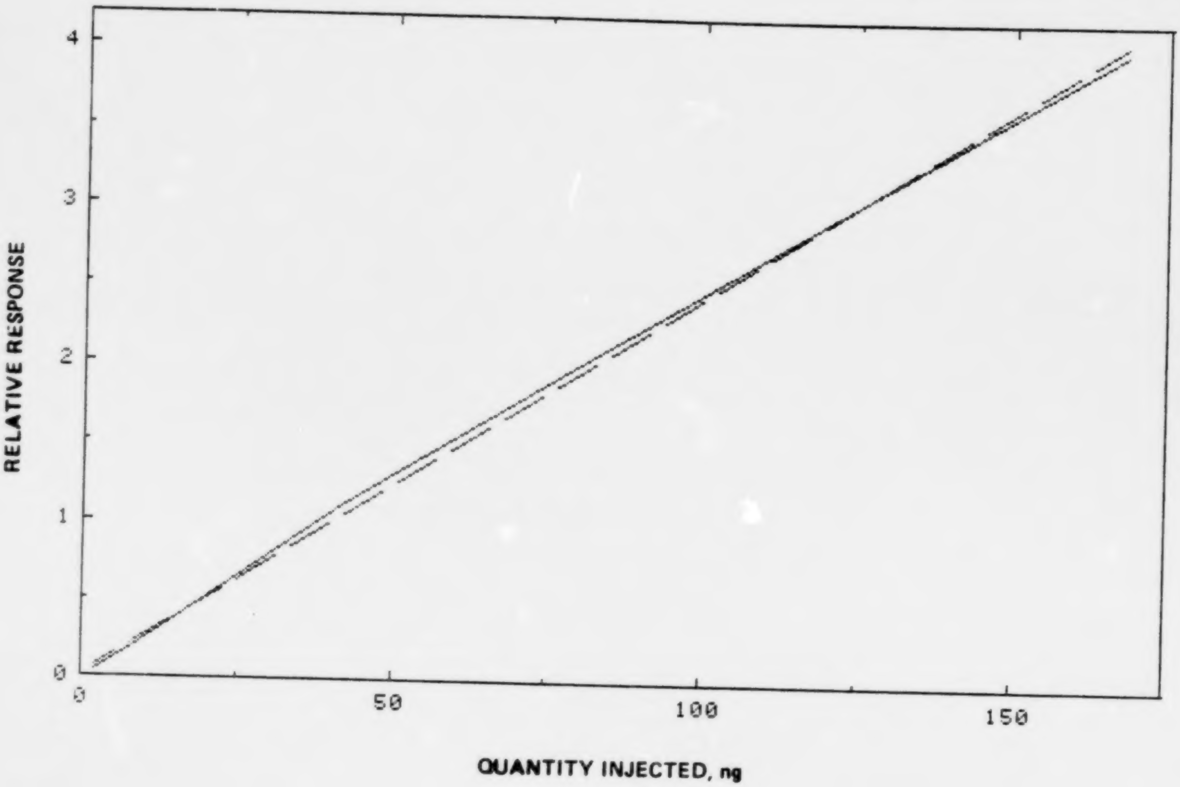


Figure A-38. Calibration curve for the determination of 1-amino-4-bromonaphthalene by GC/FID.

Range: 4.2 - 170 ng
Correlation coefficient: 0.999
Slope: 2.4×10^{-2}
y-Intercept: 2.1×10^{-2}

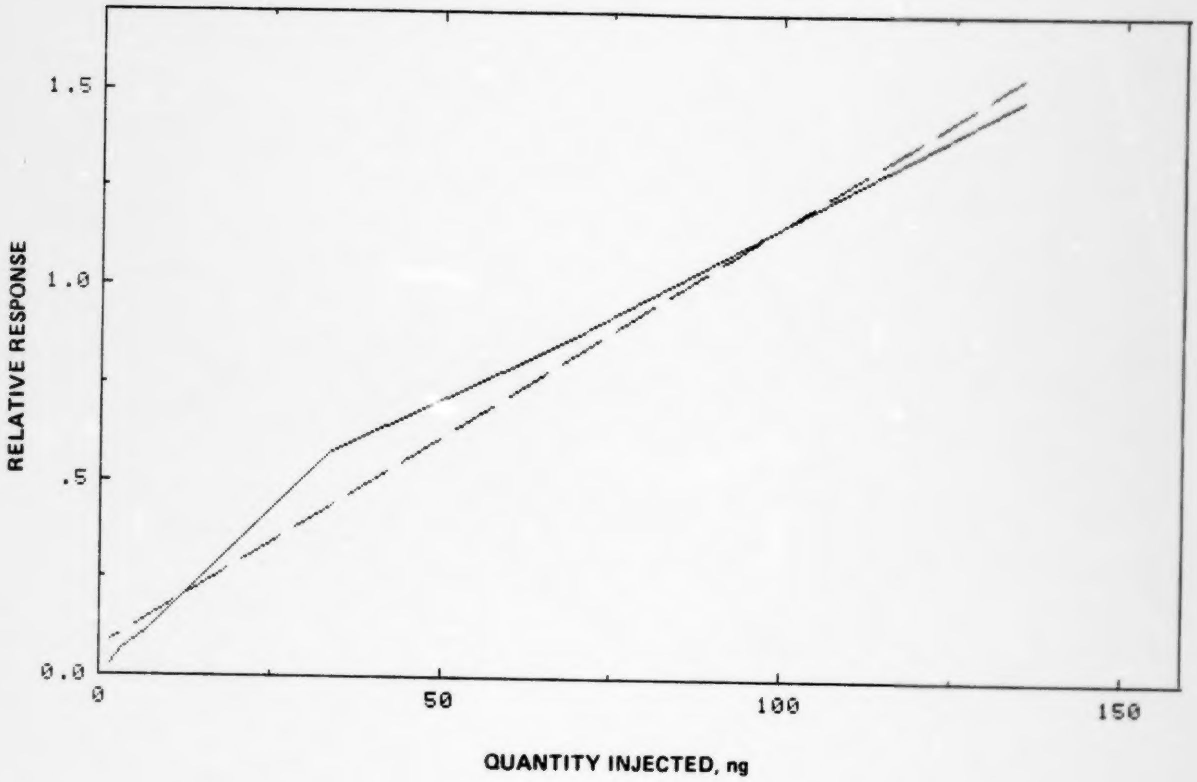


Figure A-39. Calibration curve for the determination of 2-chloroanthracene by GC/FID.

Range: 6.8 - 140 ng
Correlation coefficient: 0.991
Slope: 1.1×10^{-2}
y-Intercept: 6.9×10^{-2}

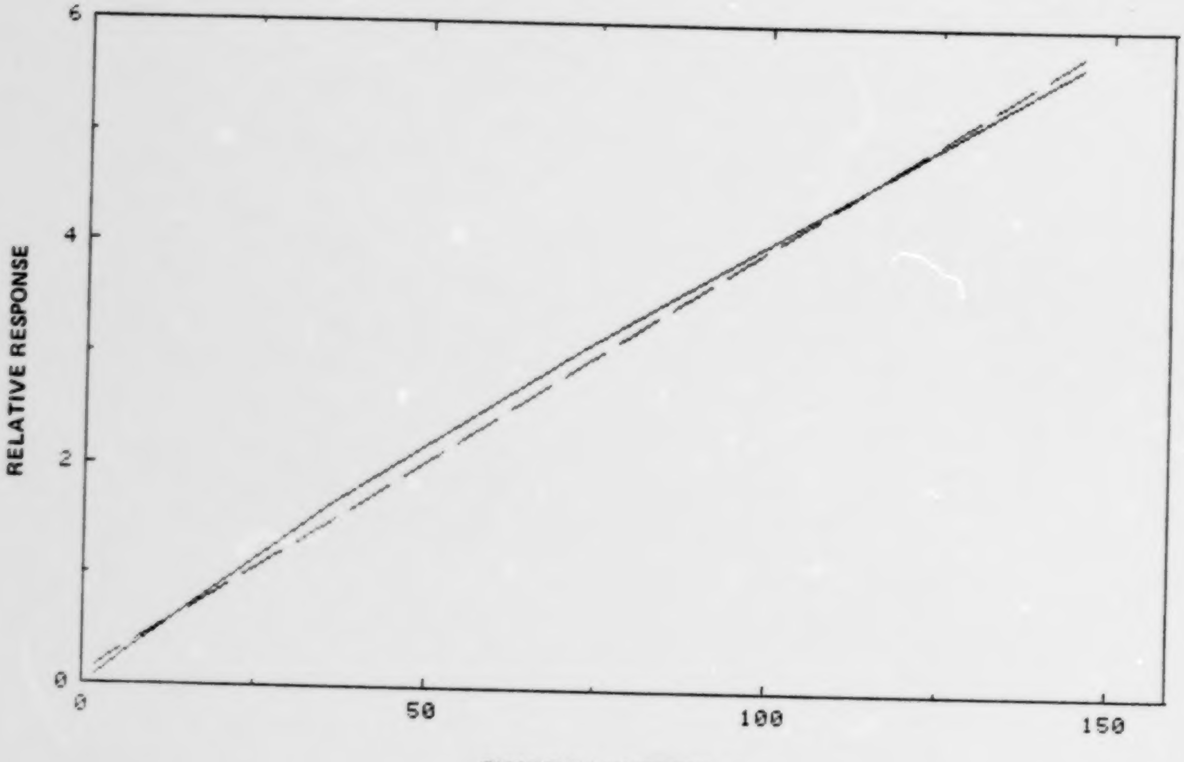


Figure A-40. Calibration curve for the determination of 9-(chloromethyl)anthracene by GC/FID.

Range: 3.6 - 150 ng
Correlation coefficient: 0.999
Slope: 3.9×10^{-2}
y-Intercept: 8.8×10^{-2}

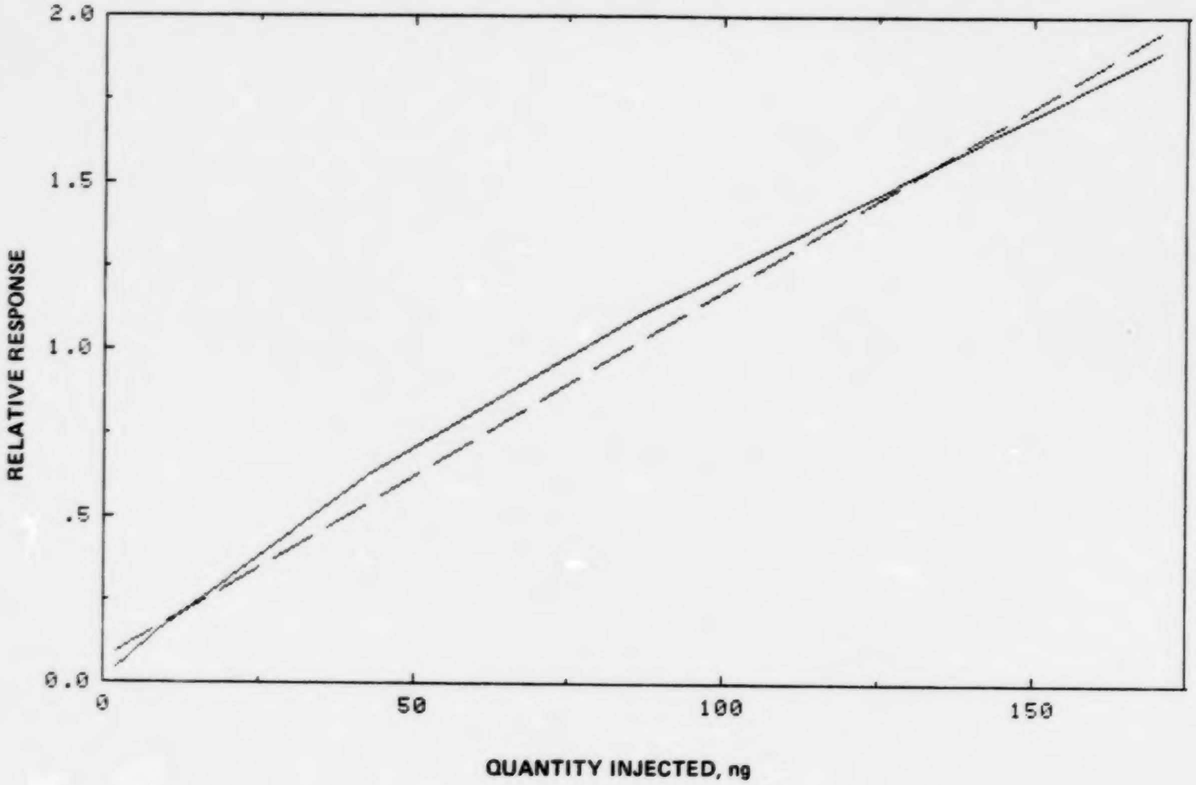


Figure A-41. Calibration curve for the determination of 9,10-dibromoanthracene by GC/FID.

Range: 8.5 - 170 ng
Correlation coefficient: 0.996
Slope: 1.1×10^{-2}
y-Intercept: 7.1×10^{-2}

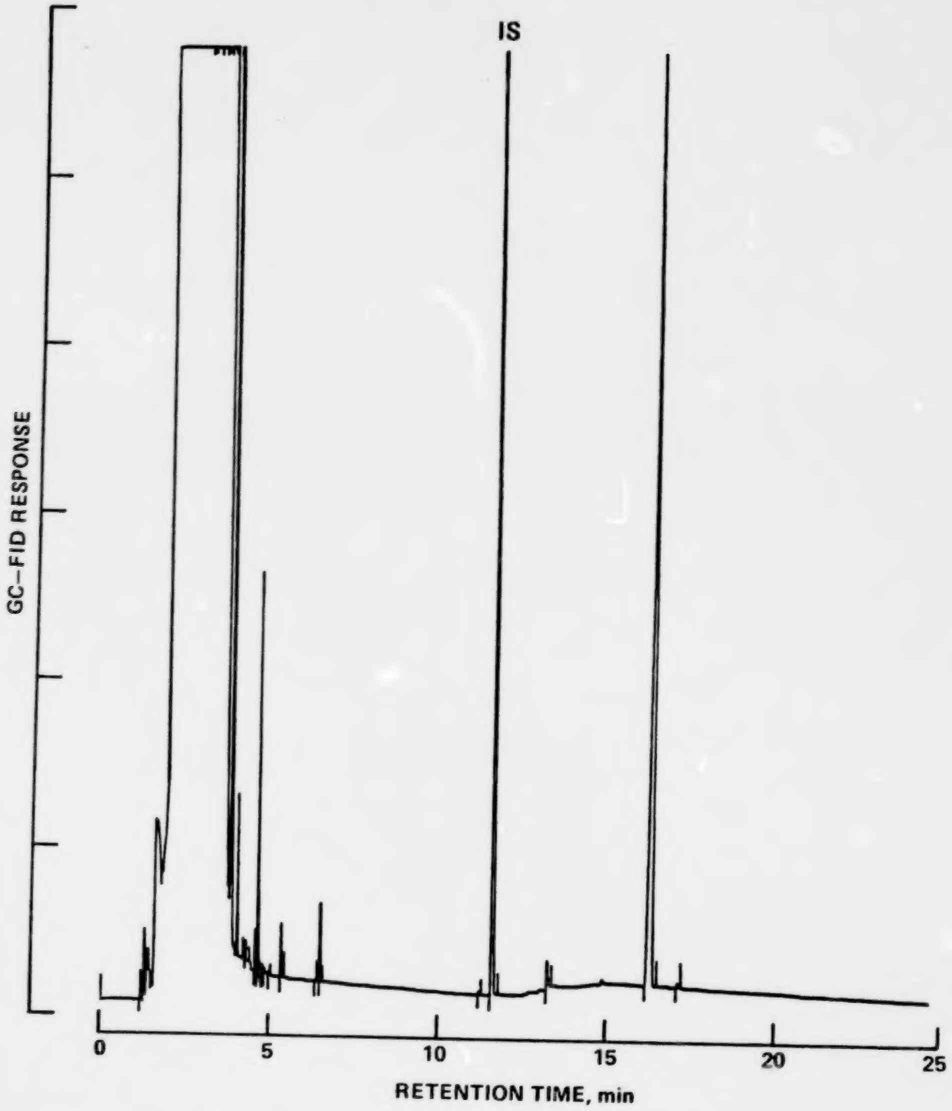


Figure A-42. Chromatogram by GC/FID for 1,2,3,4,7-pentachlorodibenzo-p-dioxin(t_R = 16.4 min).

Temperature program:
90 °C, 3 min isothermal
90 to 300 °C at 20 °C/min
300 °C, 10 min isothermal

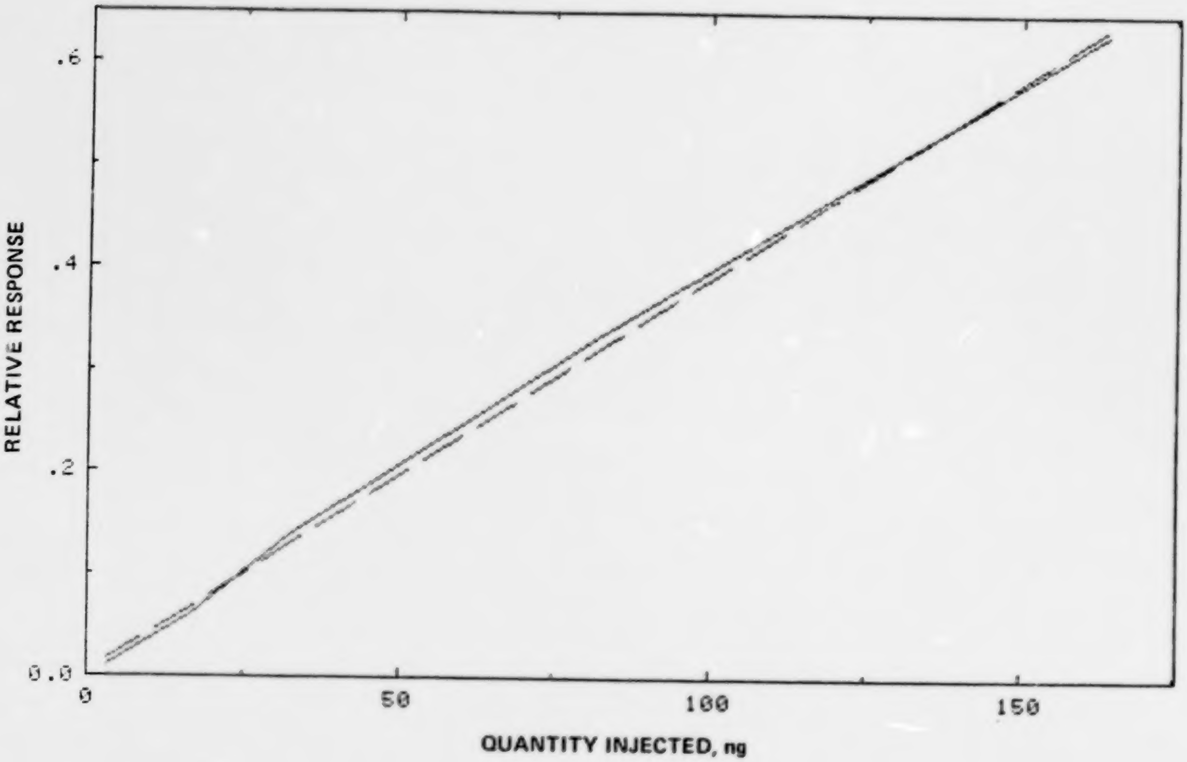


Figure A-43. Calibration curve for the determination of 1,2,3,4,7-pentachlorodibenzo-p-dioxin by GC/FID.

Range: 10 - 160 ng
Correlation coefficient: 0.999
Slope: 3.9×10^{-3}
y-Intercept: 3.6×10^{-3}

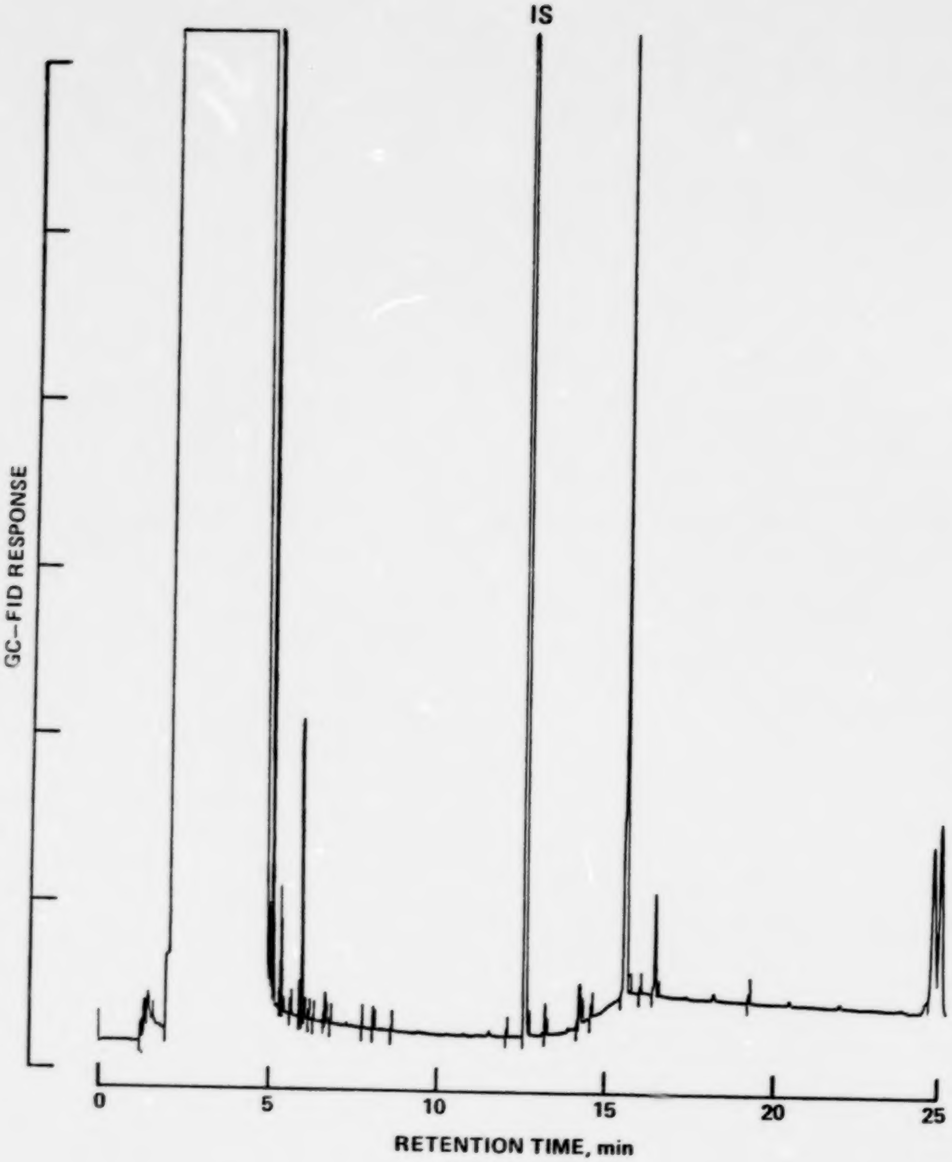


Figure A-44. Chromatogram by GC/FID for 2,3,7,8-tetrachlorodibenzofuran(t_R = 15.7 min).

Temperature program:
60 °C, 3 min isothermal
60 to 300 °C at 20 °C/min
300 °C, 10 min isothermal

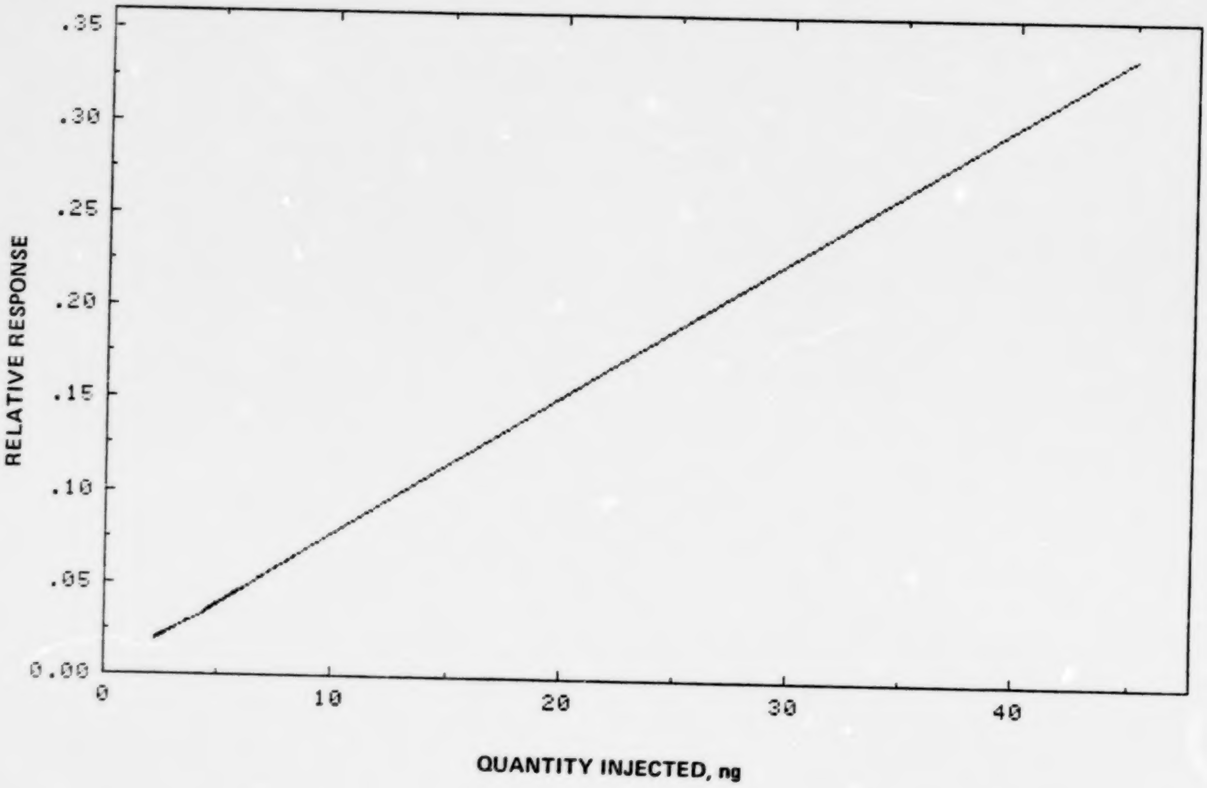


Figure A-45. Calibration curve for the determination of 2,3,7,8-tetrachlorodibenzofuran by GC/FID.

Range: 2.2 - 45 ng
Correlation coefficient: 0.999
Slope: 7.5×10^{-3}
y-Intercept: 2.2×10^{-3}

APPENDIX B

CHROMATOGRAMS, MASS SPECTRA, AND CALIBRATION CURVES FOR GC/MS

Index

Compound	Reference chromato-gram Figure No.	Reference mass spectrum Figure No.	Reference calibration curve Figure No.
1-Amino-4-bromonaphthalene	B-57	B-64	B-65
1-Amino-4-chloronaphthalene	B-57	B-60	B-61
5-Bromoacenaphthene	B-35	B-42	B-43
9-Bromoanthracene	B-35	B-50	B-51
2-Bromofluorene	B-35	B-46	B-47
9-Bromofluorene	B-57	B-62	B-63
1-Bromo-2-methylnaphthalene	B-1	B-4	B-5
1-Bromo-4-methylnaphthalene	B-35	B-40	B-41
2-(Bromomethyl)naphthalene	B-1	B-6	B-7
2-Bromonaphthalene	B-1	B-2	B-3
9-Bromophenanthrene	B-1	B-10	B-11
3-Bromoquinoline	B-35	B-38	B-39
1-Chloroanthracene	B-1	B-8	B-9
2-Chloroanthracene	B-57	B-66	B-67
9-Chloroanthracene	B-35	B-48	B-49
1-Chlorodibenzo-p-dioxin	B-16	B-17	B-18
2-Chlorodibenzo-p-dioxin	B-27	B-28	B-29
9-(Chloromethyl)anthracene	B-57	B-68	B-69
1-Chloro-8-nitronaphthalene	B-35	B-44	B-45
9,10-Dibromoanthracene	B-57	B-70	B-71
3,6-Dibromocarbazole	B-35	B-52	B-53
9,10-Dichloroanthracene	B-1	B-12	B-13
3,6-Dichlorodibenzofuran	B-32	B-33	B-34
2,3-Dichlorodibenzo-p-dioxin	B-27	B-30	B-31
2,7-Dichlorodibenzo-p-dioxin	B-16	B-19	B-20
6,9-Dichloro-2-methoxyacridine	B-1	B-14	B-15
1-Fluoronaphthalene	B-35	B-36	B-37
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	B-54	B-55	B-56
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	B-16	B-25	B-26
1-Iodonaphthalene	B-57	B-58	B-59
1,2,3,4,6,7,8,9-Octachlorodibenzofuran	B-75	B-80	B-81
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin	B-75	B-78	B-79
1,2,3,4,7-Pentachlorodibenzo-p-dioxin	B-72	B-73	B-74
2,3,7,8-Tetrachlorodibenzofuran	B-75	B-76	B-77
1,2,3,4-Tetrachlorodibenzo-p-dioxin	B-16	B-23	B-24
1,2,4-Trichlorodibenzo-p-dioxin	B-16	B-21	B-22

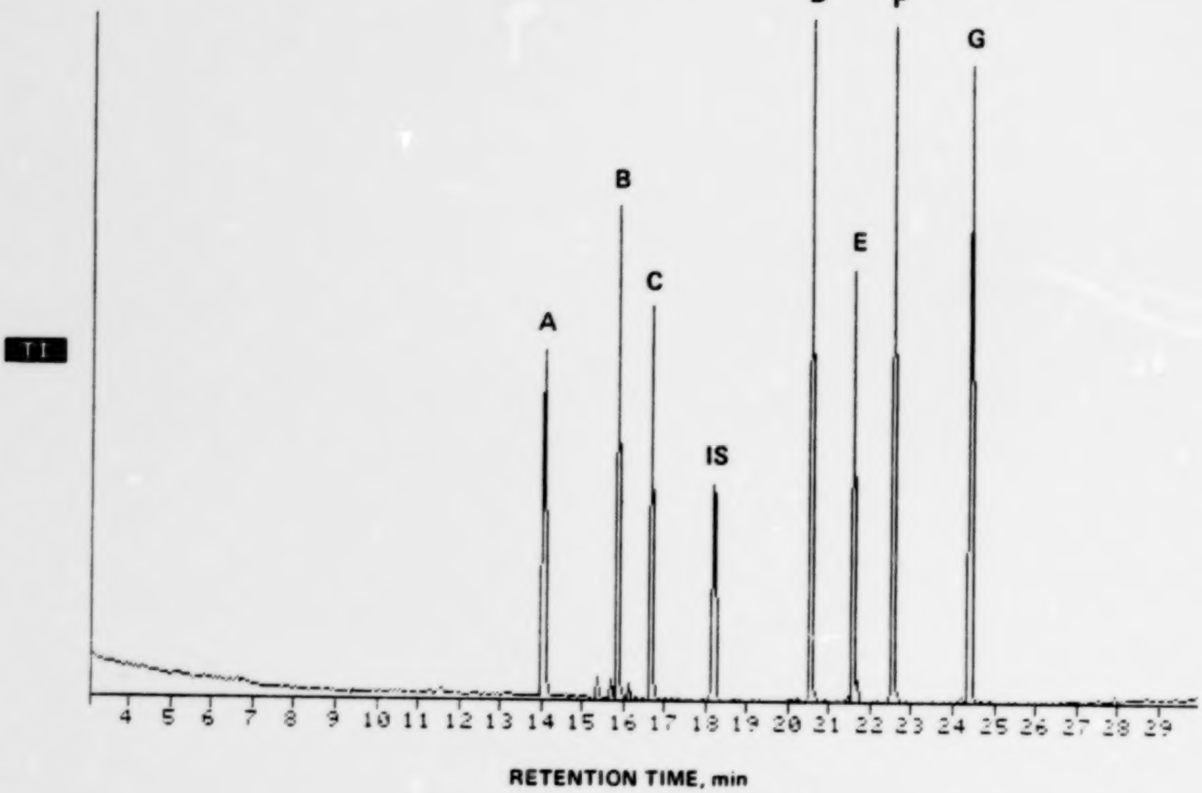


Figure B-1. Chromatogram by GC/MS for

(A) 2-bromonaphthalene(t_R = 14.1 min),
(B) 1-bromo-2-methylnaphthalene(t_R = 16.0 min),
(C) 2-(bromomethyl)naphthalene(t_R = 16.8 min),
(D) 1-chloroanthracene(t_R = 20.6 min),
(E) 9-bromophenanthrene(t_R = 21.7 min),
(F) 9,10-dichloroanthracene(t_R = 22.6 min), and
(G) 6,9-dichloro-2-methoxyacridine(t_R = 24.5 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 1 min isothermal

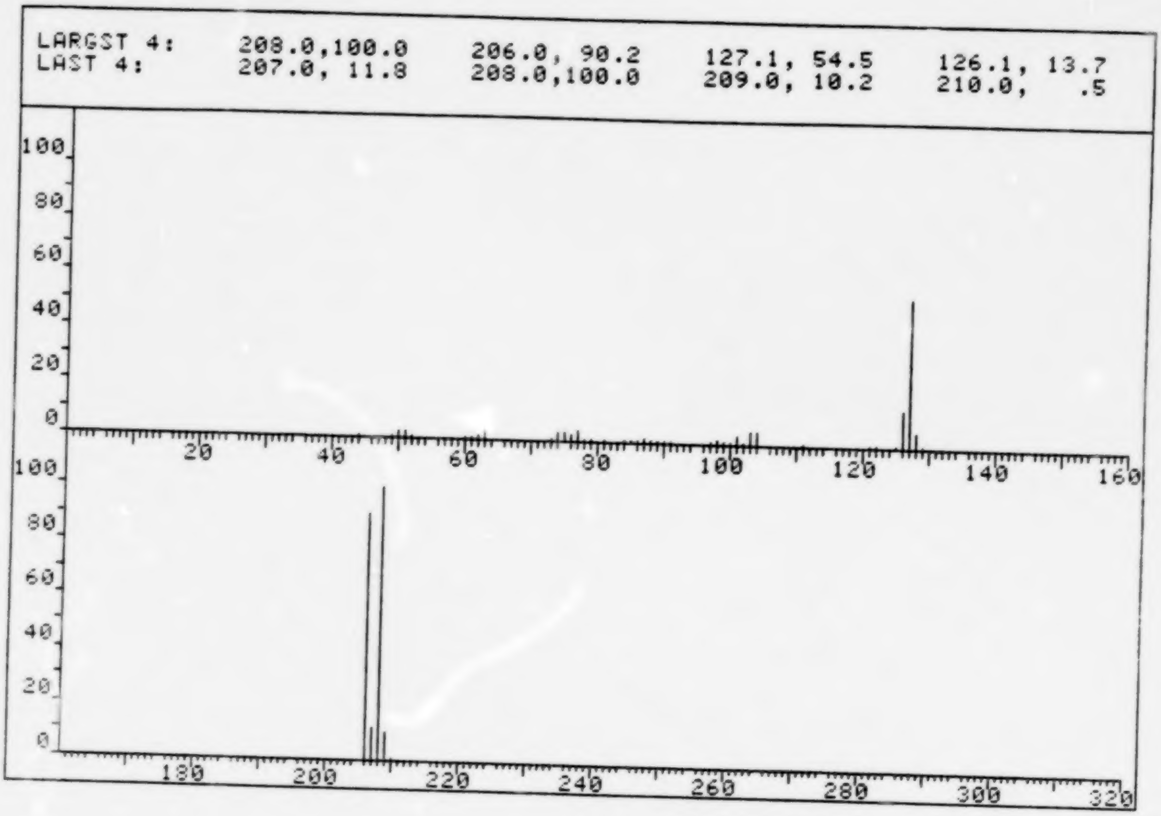


Figure B-2. Mass spectrum for 2-bromonaphthalene.

TABLE B-1. KEY IONS AND RELATIVE ABUNDANCES FOR 2-BROMONAPHTHALENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	.0	76	2.0	91	1.0	125	1.1
49	.3	77	4.2	97	.3	126	13.7
50	2.5	78	.4	98	1.6	127	54.5
51	2.1	79	.2	99	1.3	128	5.8
52	.3	81	.2	100	1.1	129	.4
60	.2	84	.1	101	3.2	206	90.2
61	.9	85	.5	102	.4	207	11.8
62	1.6	86	1.2	103	4.8	208	100.0
63	7.7	87	1.4	104	5.0	209	10.2
73	.5	88	.3	111	.2	210	.5
74	3.6	89	.2	121	.2		
75	3.8	90	.9	122	.4		

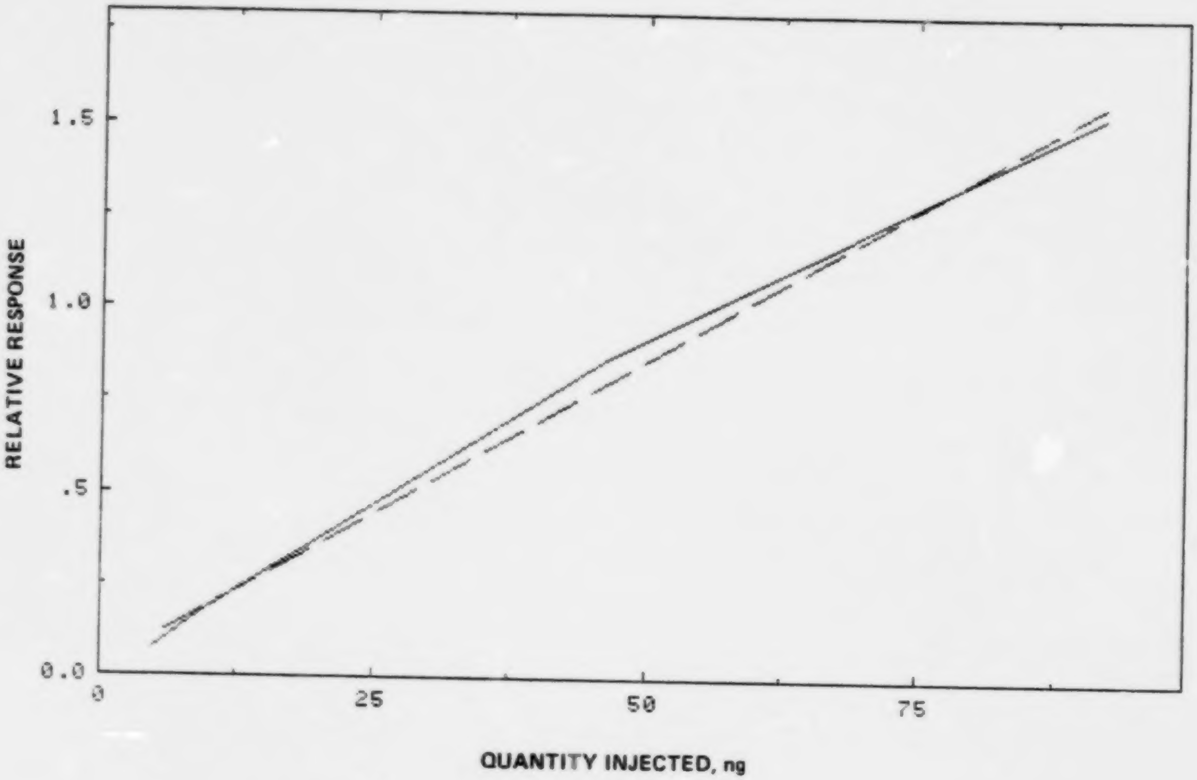
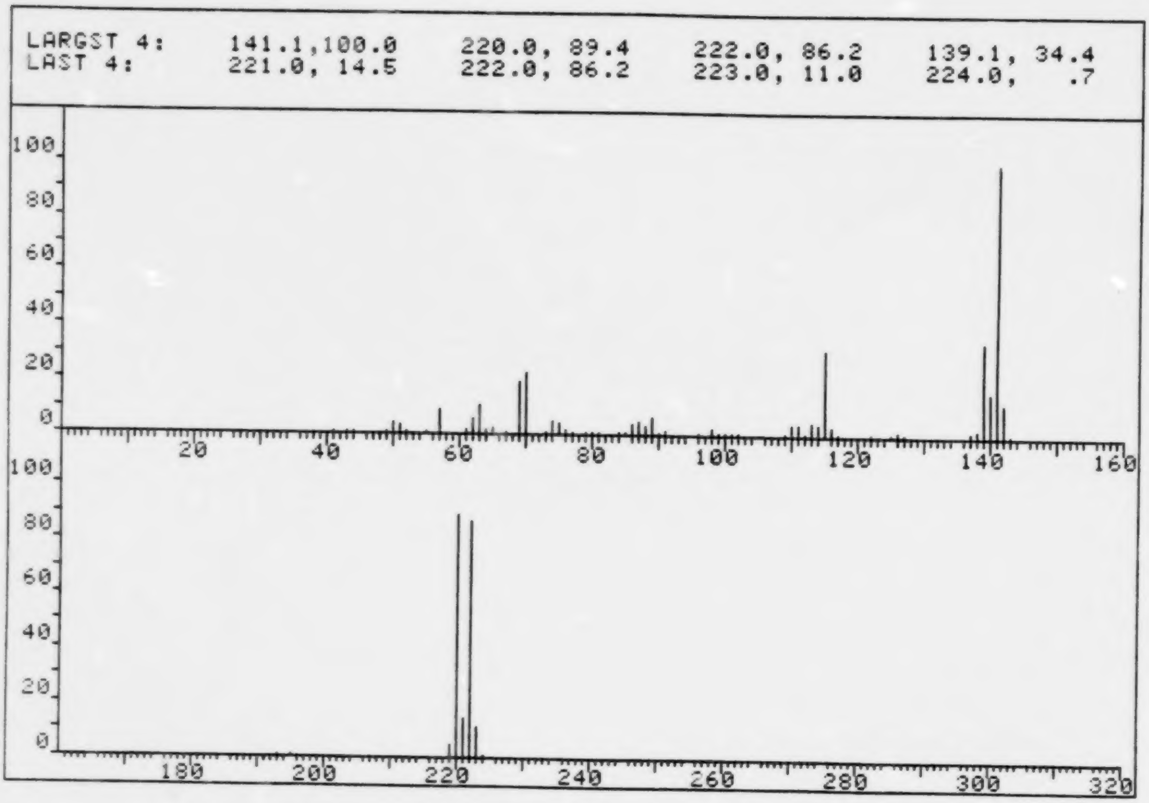


Figure B-3. Calibration curve for the determination of 2-bromonaphthalene by GC/MS.

Range: 5.0 - 90 ng
Correlation coefficient: 0.998
Slope: 1.7×10^{-2}
y-Intercept: 2.4×10^{-2}



```
LARGST 4:     141.1,100.0     220.0, 89.4     222.0, 86.2     139.1, 34.4
LAST 4:       221.0, 14.5     222.0, 86.2     223.0, 11.0     224.0,    .7
```

Figure B-4. Mass spectrum 1-bromo-2-methylnaphthalene.

TABLE B-2. KEY IONS AND RELATIVE ABUNDANCES FOR 1-BROMO-2-METHYLNAPHTHALENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	.3	73	.7	98	2.2	127	.4
43	.2	74	5.3	99	1.1	137	1.7
44	.2	75	4.2	100	.6	138	2.6
49	.5	76	2.0	101	.3	139	34.4
50	4.0	77	.8	102	1.0	140	16.5
51	3.7	79	.3	109	.5	141	100.0
52	.8	80	.2	110	4.1	142	12.3
55	1.0	81	.4	111	4.6	143	.8
57	9.1	84	.3	112	.5	193	.5
61	1.7	85	1.2	113	5.1	195	.5
62	5.4	86	3.9	114	3.9	219	4.5
63	10.9	87	4.7	115	31.8	220	89.4
64	1.5	88	3.1	116	3.1	221	14.5
65	2.2	89	6.4	117	.3	222	86.2
67	.3	90	.6	122	.3	223	11.0
69	19.2	91	1.6	125	.4	224	.7
70	22.9	96	.8	126	1.4		

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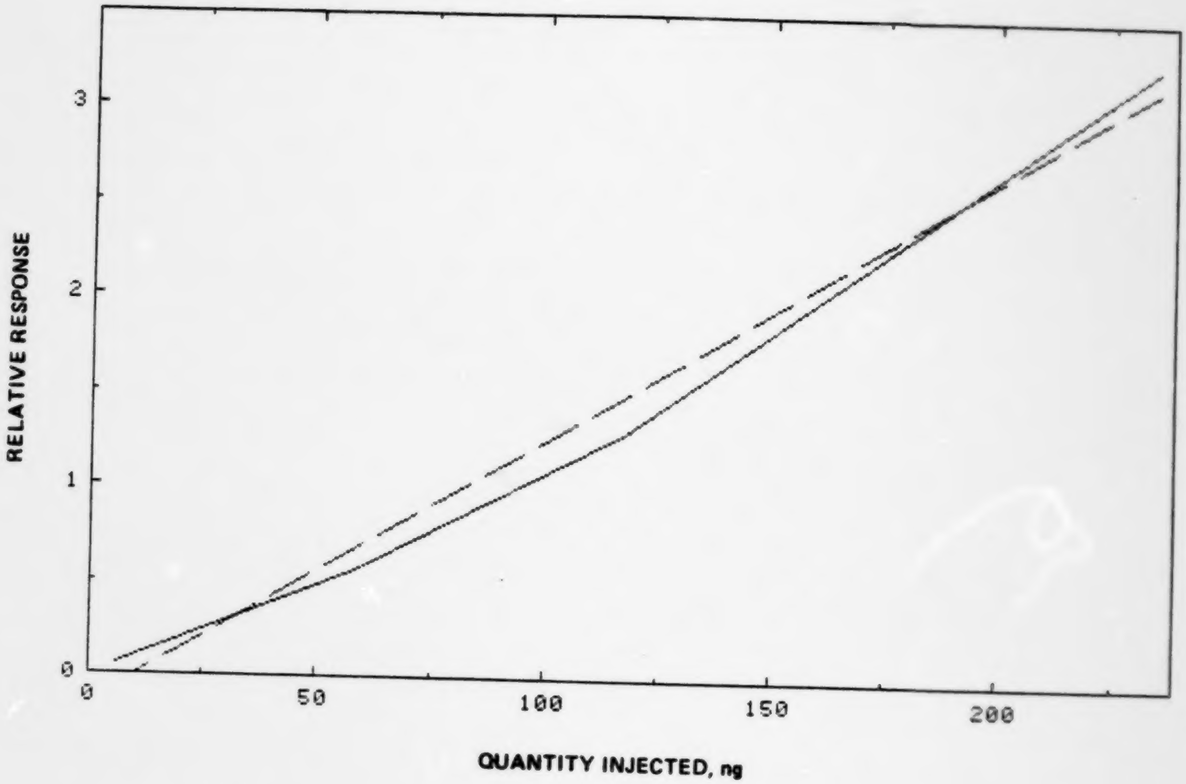
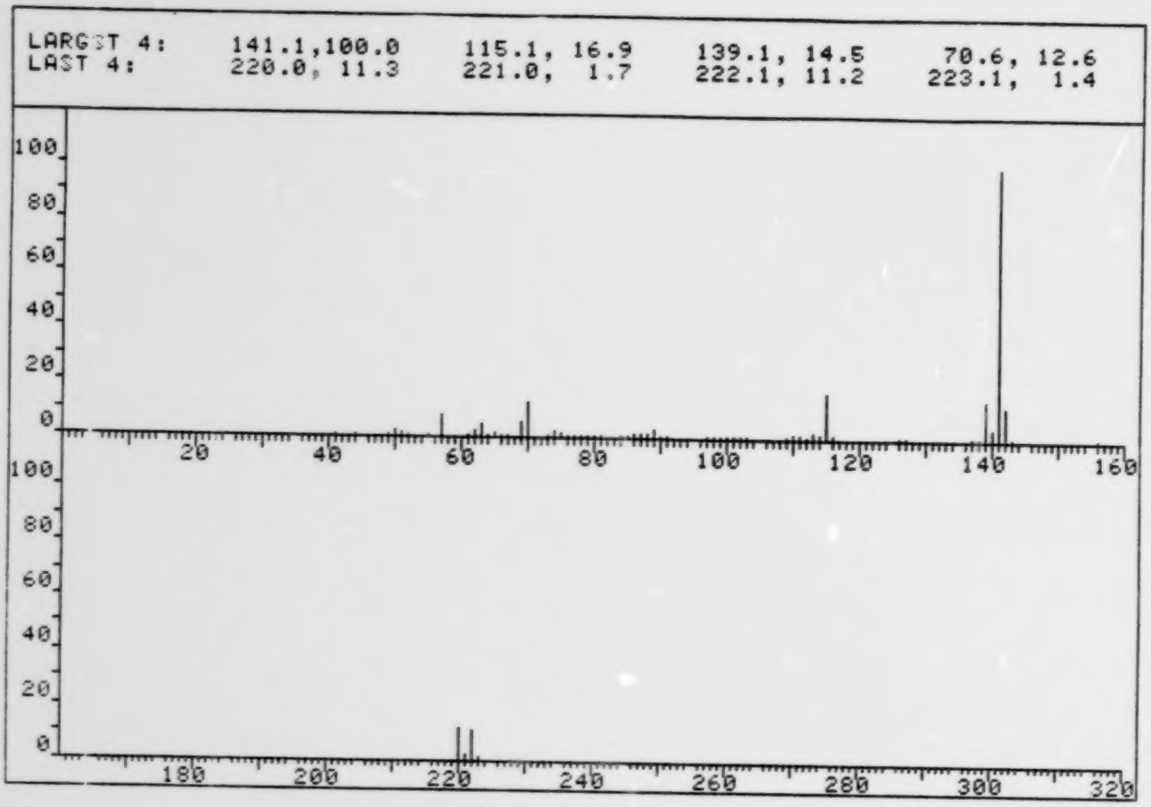


Figure B-5. Calibration curve for the determination of 1-bromo-2-methylnaphthalene by GC/MS.

Range: 50 - 240 ng
Correlation coefficient: 0.994
Slope: 1.4 x 10^{-2}
y-Intercept: -1.4 x 10^{-1}



```
LARGST 4:    141.1,100.0     115.1, 16.9     139.1, 14.5      70.6, 12.6
LAST 4:      220.0, 11.3     221.0,  1.7     222.1, 11.2     223.1,  1.4
```

Figure B-6. Mass spectrum for 2-(bromomethyl)naphthalene.

TABLE B-3. KEY IONS AND RELATIVE ABUNDANCES FOR 2-(BROMOMETHYL)NAPHTHALENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	.2	70	12.6	90	.4	115	16.9
44	.0	73	.3	91	1.0	116	1.7
49	.3	74	2.2	97	.1	126	.4
50	2.3	75	1.7	97	.1	127	.3
51	2.0	76	1.1	98	.7	137	.5
52	.5	77	.5	99	.3	138	.8
55	.3	78	.2	100	.2	139	14.5
57	8.2	79	.6	101	.1	140	4.2
61	.7	80	.2	102	.7	141	100.0
62	2.6	81	.6	103	.1	142	12.1
63	5.0	84	.2	109	.2	143	.7
64	.8	85	.4	110	1.7	156	.2
65	1.3	86	1.6	111	1.9	220	11.3
66	.1	87	2.0	112	.2	221	1.7
67	.1	88	1.4	113	2.0	222	11.2
69	5.6	89	3.1	114	1.6	223	1.4

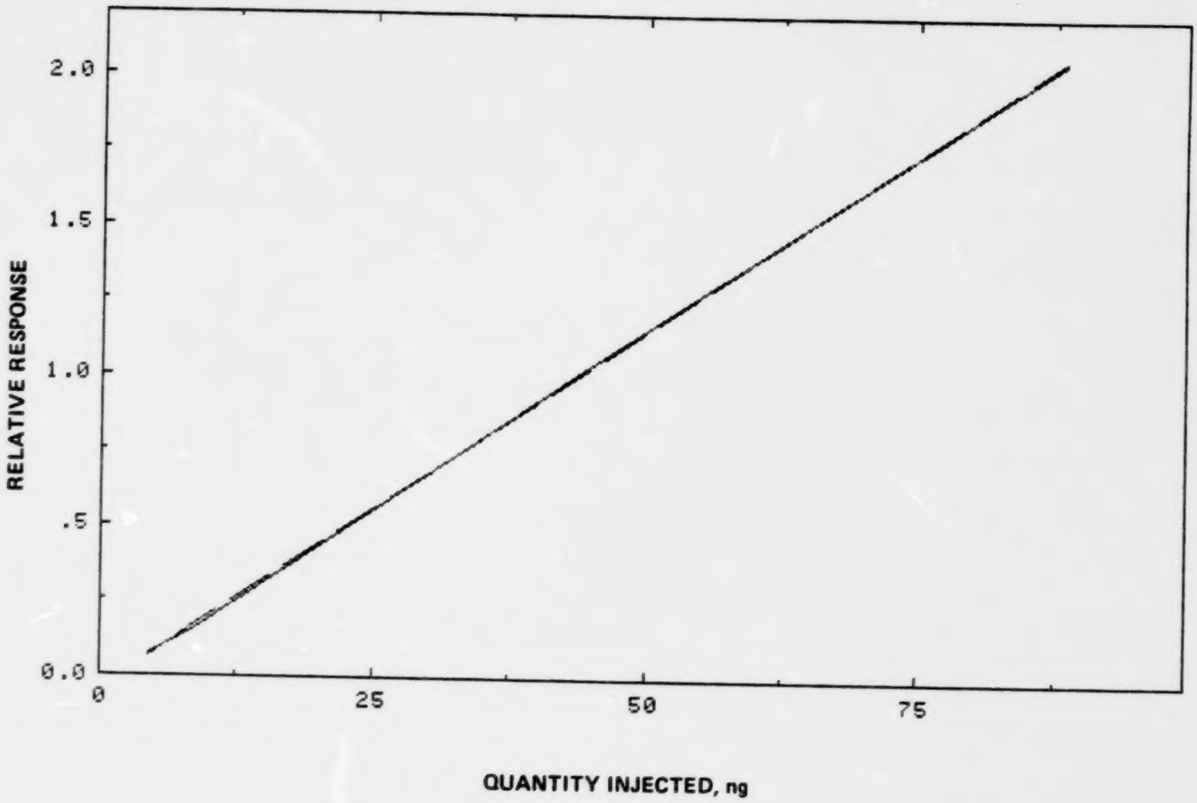
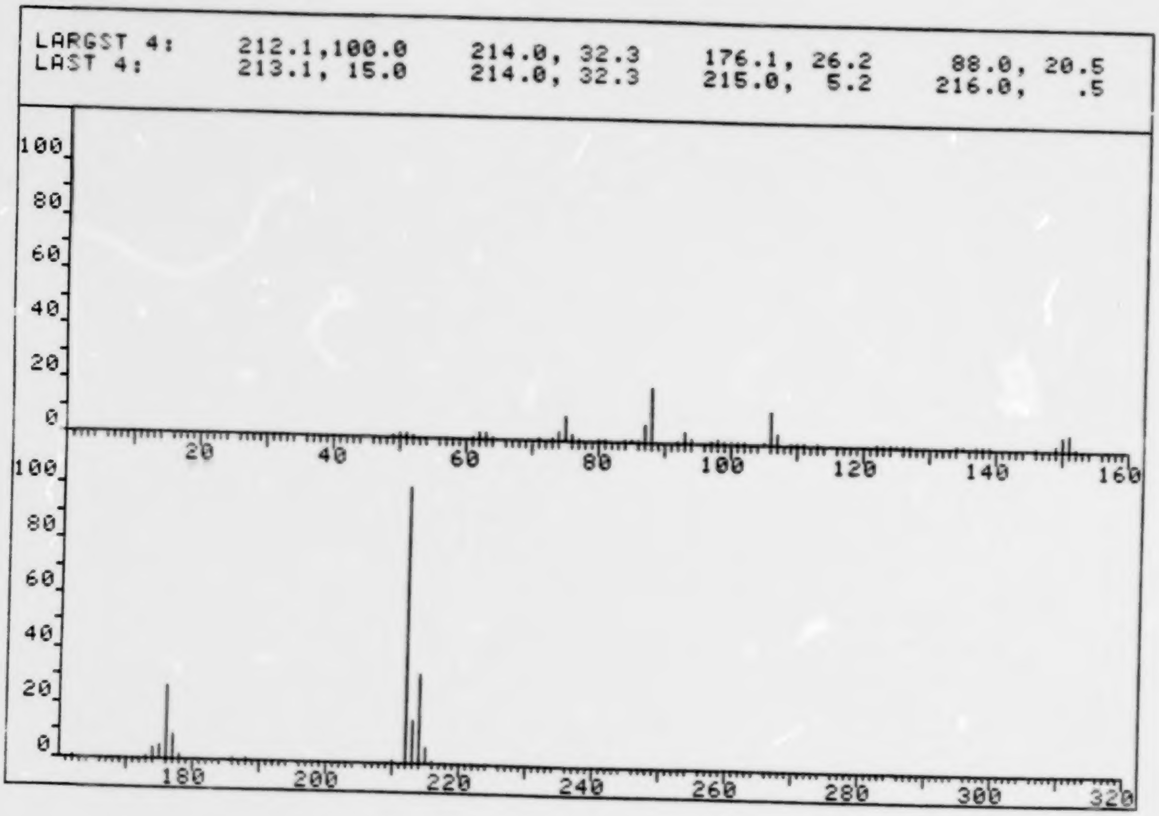


Figure B-7. Calibration curve for the determination of 2-(bromomethyl)naphthalene.

Range: 4.0 - 90 ng
Correlation coefficient: 0.999
Slope: 2.4×10^{-2}
y-Intercept: -3.9×10^{-2}



```
LARGST 4:     212.1,100.0    214.0, 32.3    176.1, 26.2     88.0, 20.5
LAST 4:       213.1, 15.0    214.0, 32.3    215.0,  5.2    216.0,    .5
```

Figure B-8. Mass spectrum for 1-chloroanthracene.

TABLE B-4. KEY IONS AND RELATIVE ABUNDANCES FOR 1-CHLOROANTHRACENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
49	.2	85	.5	110	.5	151	5.9
50	1.6	86	1.3	111	.5	152	.9
51	1.4	87	6.2	113	.2	162	.2
52	.3	88	20.5	122	.5	173	.4
61	.7	92	.3	123	.5	174	3.7
62	2.3	93	4.5	124	.3	175	4.6
63	2.1	94	1.7	125	.6	176	26.2
64	.2	97	.2	126	1.2	177	8.8
71	.4	98	1.6	127	.6	178	1.2
73	.6	99	1.2	134	.2	186	.4
74	3.4	100	.5	135	.2	188	.2
75	9.0	101	.4	137	.3	210	.4
76	2.5	102	.2	138	.3	212	100.0
77	.6	105	.6	139	.2	213	15.0
80	.4	106	11.7	146	.2	214	32.3
81	.3	107	4.2	149	1.4	215	5.2
84	.2	109	.2	150	4.9	216	.5

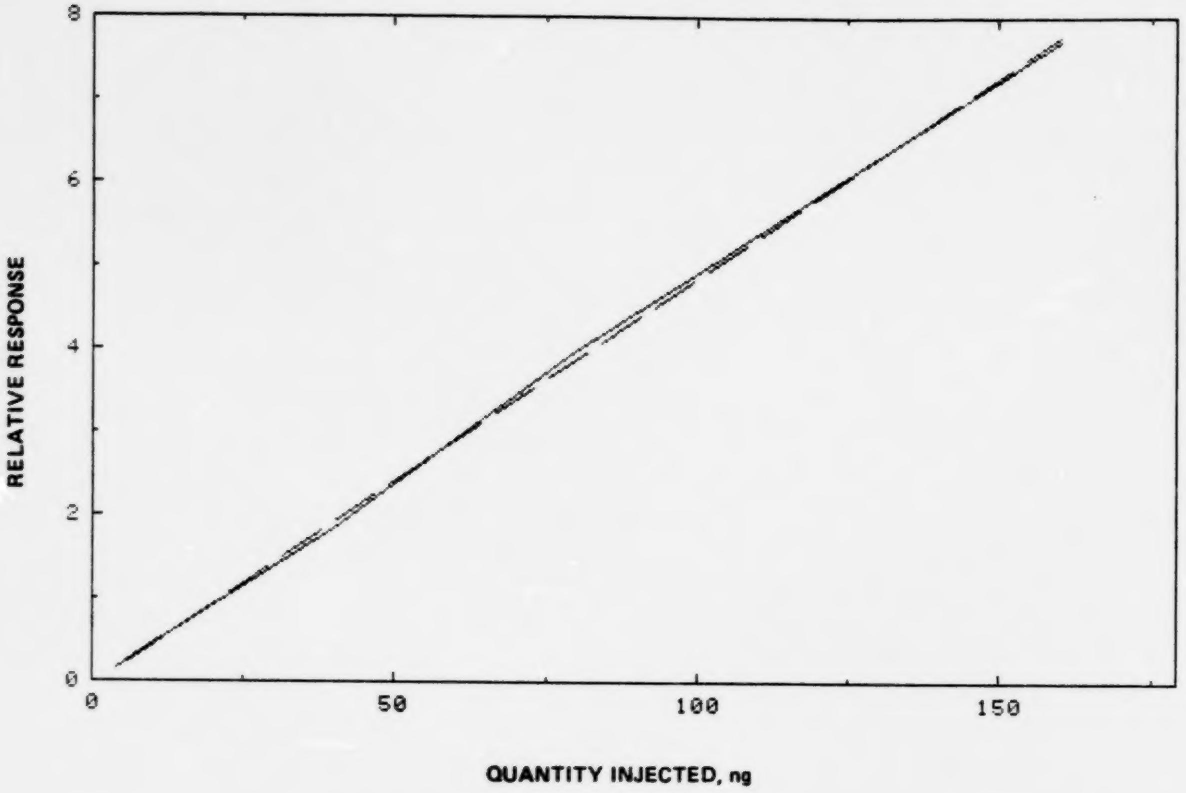
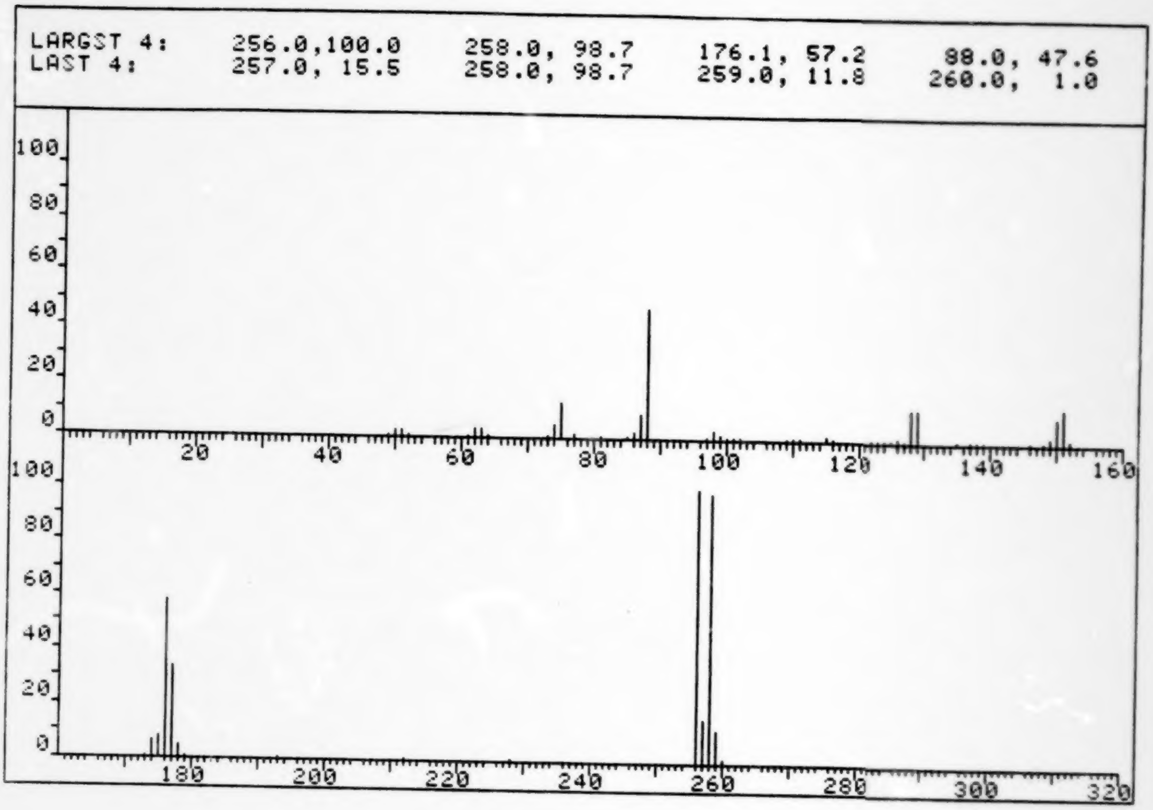


Figure B-9. Calibration curve for the determination of 1-chloroanthracene by GC/MS.

Range: 4.0 - 160 ng
Correlation coefficient: 0.999
Slope: 4.9×10^{-2}
y-Intercept: -3.5×10^{-2}



```
LARGST 4:     256.0,100.0     258.0, 98.7     176.1, 57.2      88.0, 47.6
LAST 4:       257.0, 15.5     258.0, 98.7     259.0, 11.8     260.0,  1.0
```

Figure B-10. Mass spectrum for 9-bromophenanthrene.

TABLE B-5. KEY IONS AND RELATIVE ABUNDANCES FOR 9-BROMOPHENANTHRENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
49	.3	88	47.6	126	1.7	175	8.0
50	2.3	97	.3	127	1.2	176	57.2
51	2.1	98	2.8	128	12.1	177	33.6
52	.4	99	1.9	129	11.9	178	4.9
61	1.1	100	.7	135	.3	179	.4
62	3.5	101	.7	137	.4	193	.6
63	2.8	102	.4	138	.3	212	.3
64	.3	109	.4	139	.3	228	.4
73	.7	110	.9	146	.3	256	100.0
74	5.1	111	1.0	148	.3	257	15.5
75	12.6	115	1.4	149	2.4	258	98.7
77	1.4	116	1.1	150	10.0	259	11.8
81	.3	122	1.0	151	12.7	260	1.0
85	.9	123	.8	152	1.7		
86	2.3	124	.5	173	.5		
87	9.0	125	.9	174	6.4		

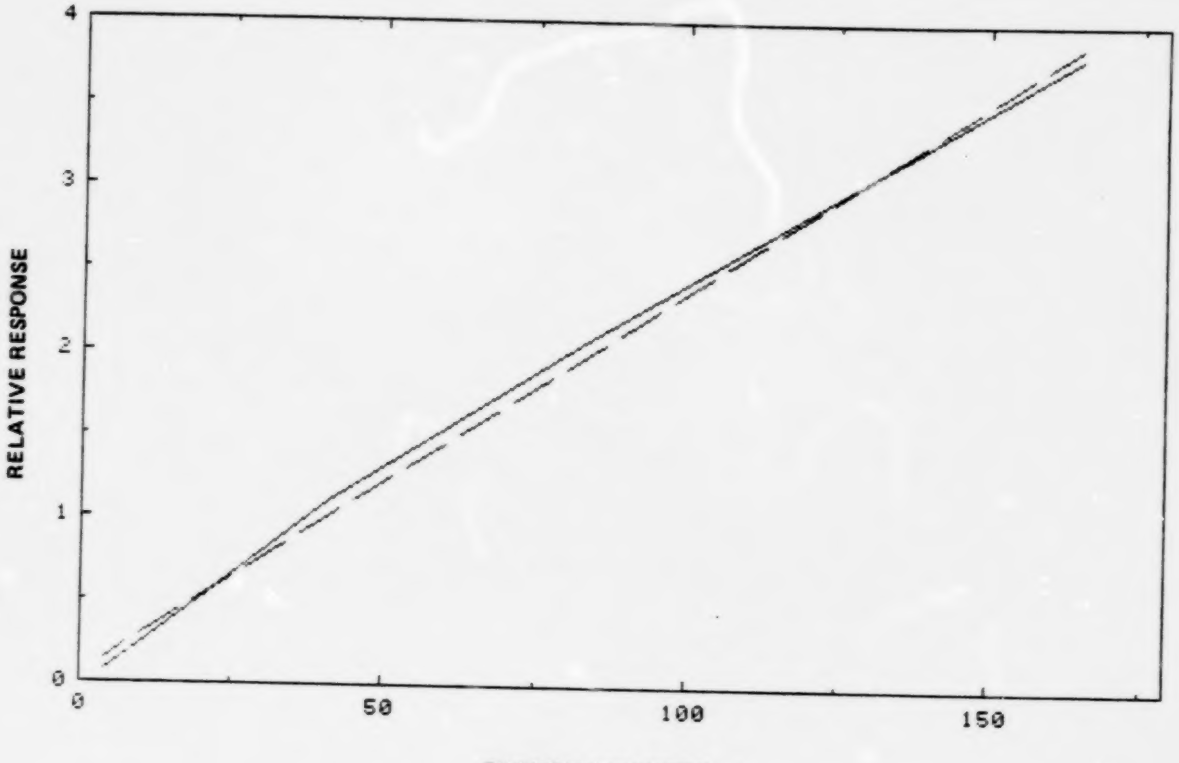
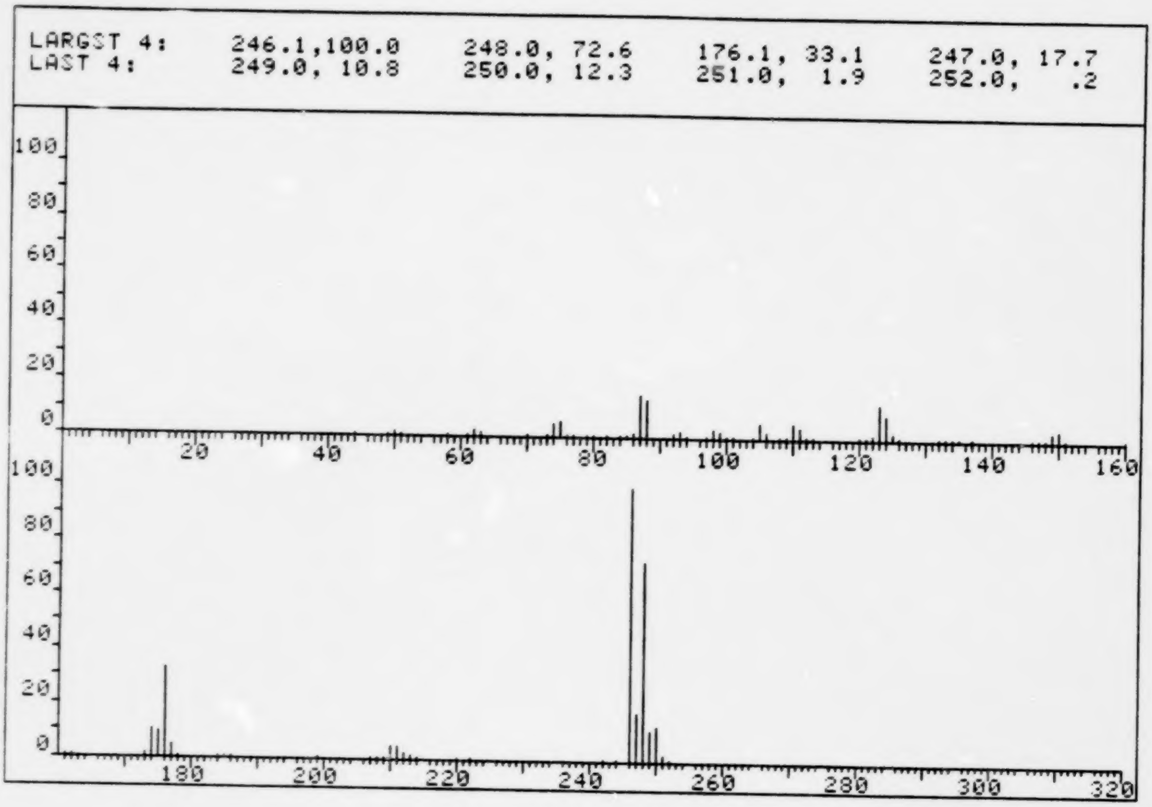


Figure B-11. Calibration curve for the determination of 9-bromophenanthrene by GC/MS.

Range: 8.0 - 160 ng
Correlation coefficient: 0.998
Slope: 2.3×10^{-2}
y-Intercept: 5.4×10^{-2}



```
LARGST 4:     246.1,100.0     248.0, 72.6     176.1, 33.1     247.0, 17.7
LAST 4:       249.0, 10.8     250.0, 12.3     251.0,  1.9     252.0,    .2
```

Figure B-12. Mass spectrum for 9,10-dichloroanthracene.

TABLE B-6. KEY IONS AND RELATIVE ABUNDANCES FOR 9,10-DICHLOROANTHRACENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	.1	93	2.7	132	.2	199	.2
49	.2	94	.7	133	.2	207	.2
50	1.4	97	.6	134	.4	208	.2
51	1.1	98	3.1	135	.5	209	.3
58	.2	99	2.1	137	.4	210	4.6
61	1.0	100	.7	146	.5	211	4.4
62	2.2	101	.3	147	.4	212	2.2
63	1.6	104	.5	148	.4	213	1.5
70	.3	105	5.6	149	2.9	214	.4
73	1.2	106	2.2	150	4.0	220	.5
74	5.1	108	.3	151	.8	222	.3
75	5.8	109	.6	160	.3	242	.2
76	1.0	110	6.0	161	.2	244	.2
77	.4	111	4.4	162	.3	246	100.0
79	.3	112	.9	173	1.0	247	17.7
80	.3	113	.2	174	9.8	248	72.6
82	.3	120	.3	175	9.6	249	10.8
84	.3	121	.3	176	33.1	250	12.3
85	.9	122	1.6	177	4.8	251	1.9
86	1.9	123	12.8	178	.4	252	.2
87	15.2	124	9.1	184	.4		
88	13.6	125	2.4	185	.3		
92	1.9	126	.9	186	.4		

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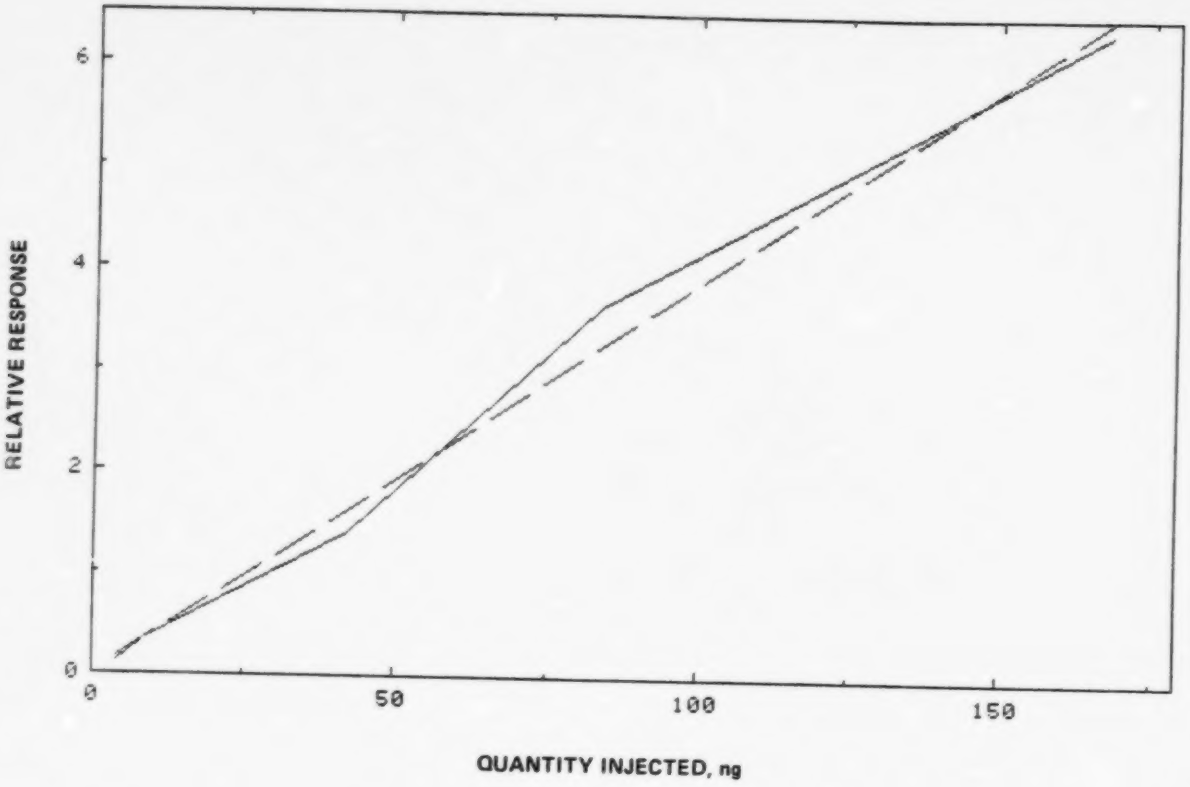


Figure B-13. Calibration curve for the determination of 9,10-dichloroanthracene by GC/MS.

Range: 4.0 - 170 ng
Correlation coefficient: 0.996
Slope: 3.9×10^{-2}
y-Intercept: 9.4×10^{-3}

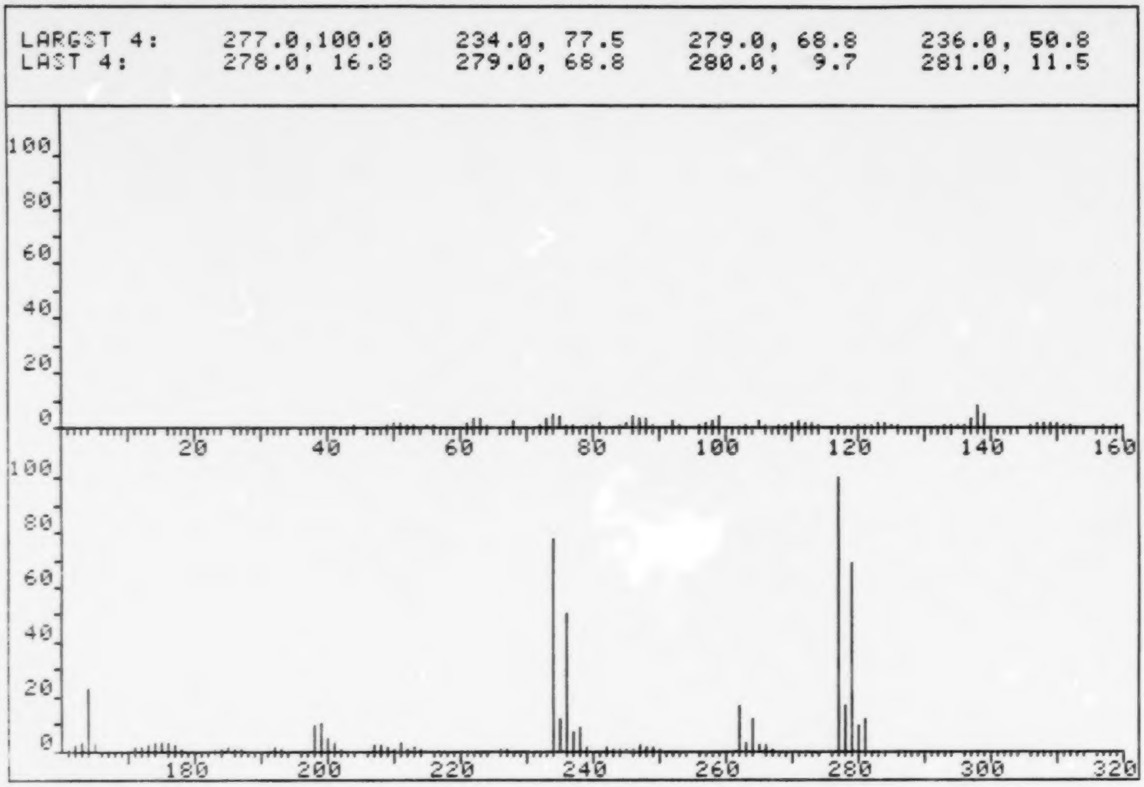


Figure B-14. Mass spectrum for 6,9-dichloro-2-methoxyacridine.

TABLE B-7. KEY IONS AND RELATIVE ABUNDANCES FOR 6,9-DICHLORO-2-METHOXYACRIDINE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	.1	99	4.0	157	.3	212	.8
49	.5	103	.3	159	.4	213	1.6
50	1.7	105	2.0	160	.4	214	.6
51	1.5	106	1.2	161	.6	226	.5
52	1.2	108	.4	162	1.9	227	.6
53	.5	109	.9	163	3.4	234	77.5
55	1.0	110	1.7	164	23.1	235	11.4
56	.6	111	2.4	165	3.3	236	50.8
61	1.5	112	1.5	171	1.1	237	6.5
62	3.3	113	1.5	172	1.5	238	8.6
63	3.4	114	.8	173	1.8	239	1.2
64	1.1	117	.6	174	2.6	242	1.6
68	2.2	120	.3	175	2.5	243	.4
72	.4	121	.7	176	2.6	244	.5
73	3.5	122	1.1	177	1.7	245	.3
74	5.2	123	1.7	178	.7	246	.4
75	4.1	124	1.4	183	.8	247	1.7
76	1.0	125	.8	184	.6	248	1.3
77	.4	126	.6	185	1.4	249	1.2
79	.4	133	.4	186	.5	250	.6
80	1.1	134	.4	187	.5	262	16.2
81	1.6	135	.8	192	1.5	263	2.8
84	.6	136	1.0	193	.3	264	11.9
85	1.5	137	3.7	198	9.3	265	1.7
86	4.6	138	8.3	199	10.0	266	1.9
87	3.6	139	5.0	200	4.6	267	.4
88	3.0	146	.3	201	3.0	277	100.0
89	1.2	147	2.0	202	.5	278	16.8
92	2.2	148	1.3	207	2.0	279	68.8
93	.9	149	1.7	208	2.0	280	9.7
96	.5	150	1.6	209	1.0	281	11.5
97	1.7	151	.8	210	.4		
98	2.6	152	.4	211	3.4		

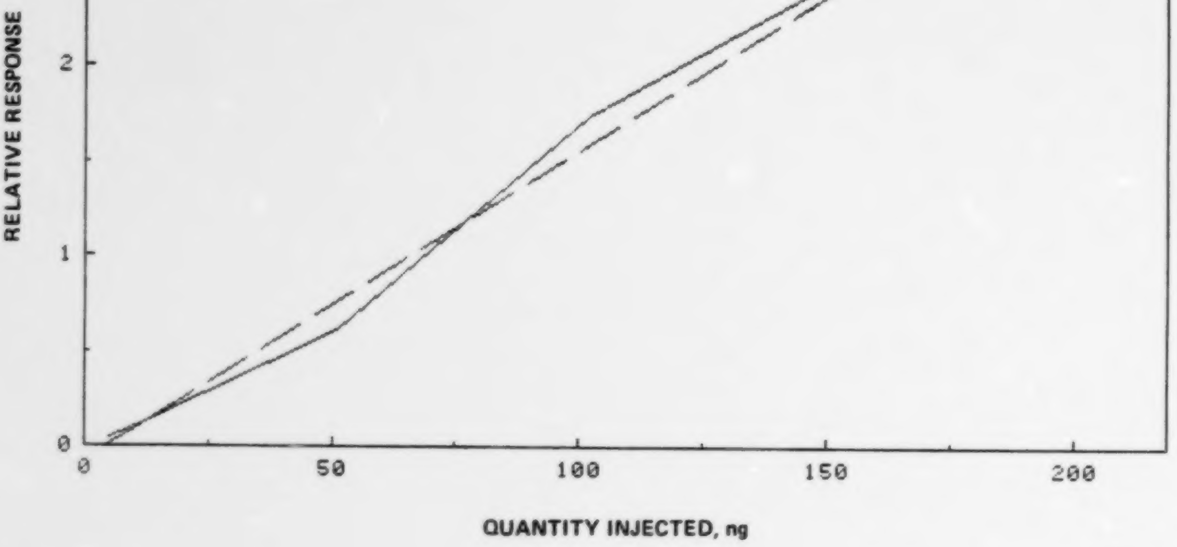


Figure B-15. Calibration curve for the determination of 6,9-dichloro-2-methoxyacridine by GC/MS.

Range: 10 - 200 ng
Correlation coefficient: 0.997
Slope: 1.6×10^{-2}
y-Intercept: -3.9×10^{-2}

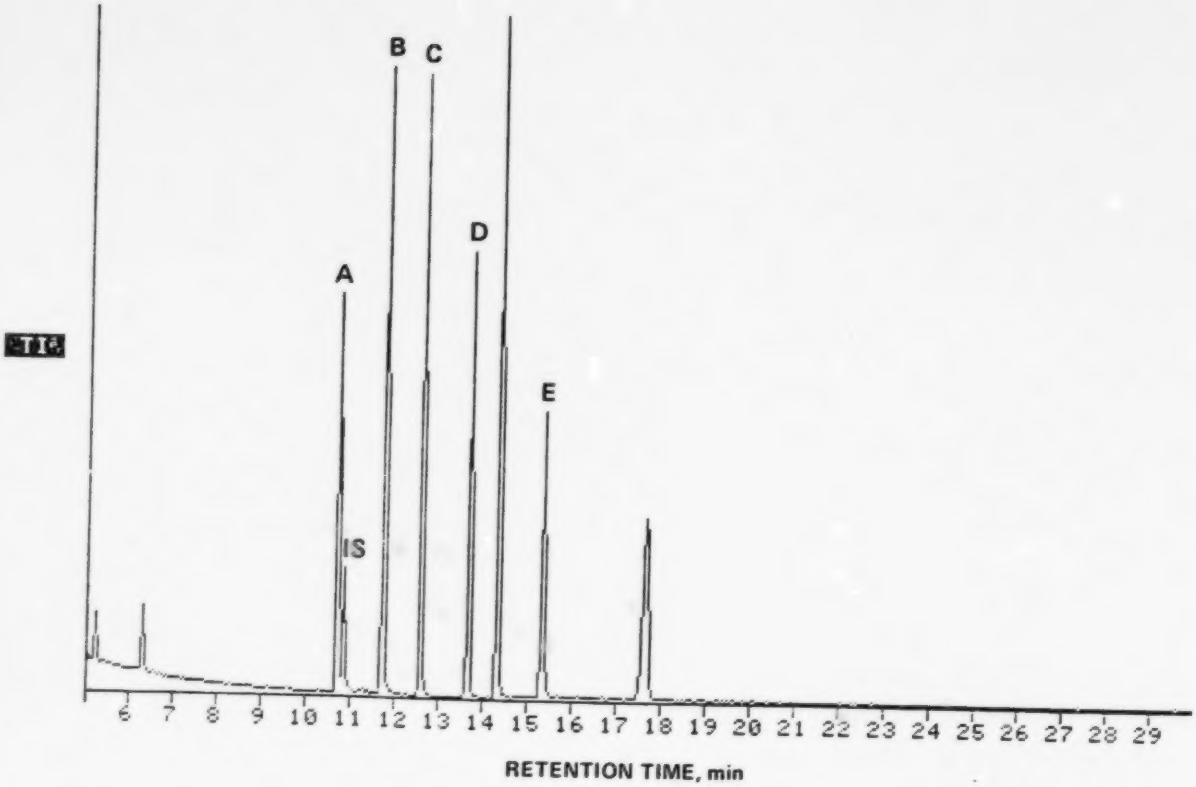
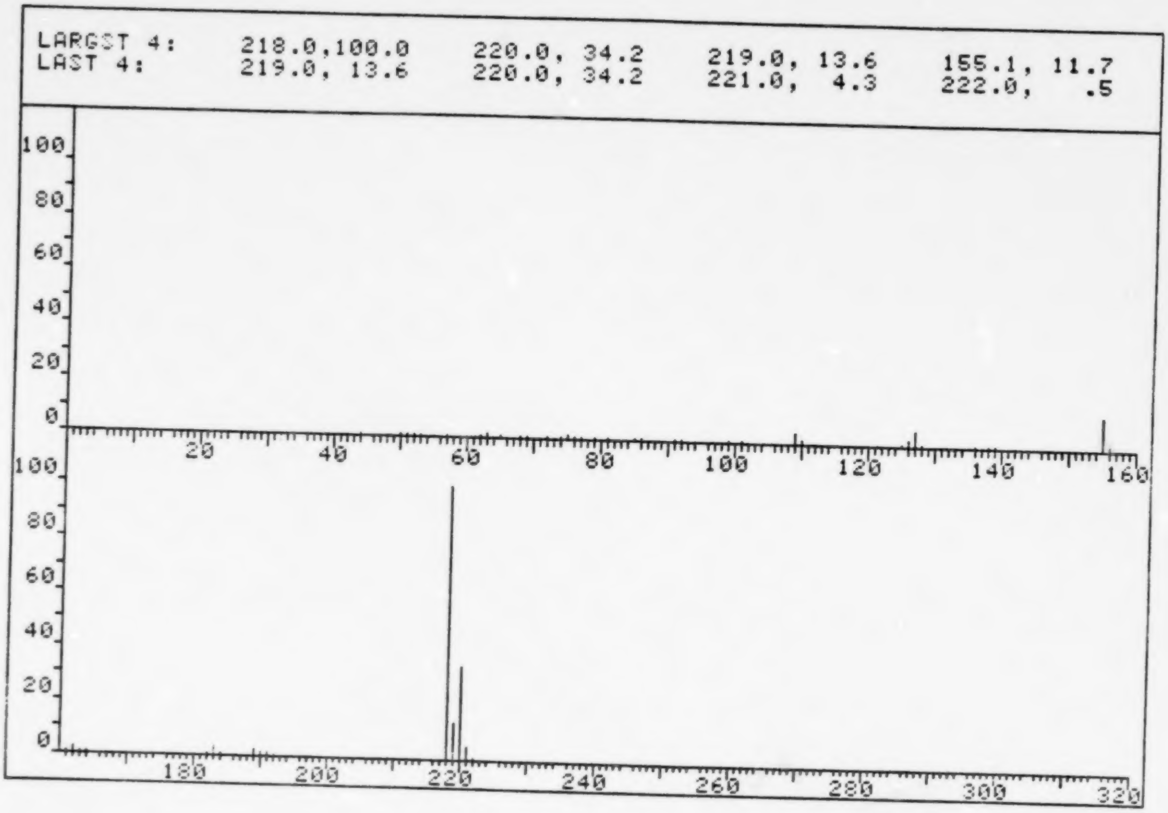


Figure B-16. Chromatogram by GC/MS for

(A) 1-chlorodibenzo-p-dioxin(t_R = 10.8 min),
(B) 2,7-dichlorodibenzo-p-dioxin(t_R = 11.7 min),
(C) 1,2,4-trichlorodibenzo-p-dioxin(t_R = 12.6 min),
(D) 1,2,3,4-tetrachlorodibenzo-p-dioxin(t_R = 13.7 min), and
(E) 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin(t_R = 15.4 min).

Temperature program:
60 °C, 3 min isothermal
60 to 300 °C at 20 °C/min
300 °C, 10 min isothermal



```
LARGST 4:    218.0,100.0    220.0, 34.2    219.0, 13.6    155.1, 11.7
LAST 4:      219.0, 13.6    220.0, 34.2    221.0,  4.3    222.0,    .5
```

Figure B-17. Mass spectrum for 1-chlorodibenzo-p-dioxin.

TABLE B-8. KEY IONS AND RELATIVE ABUNDANCES FOR 1-CHLORODIBENZO-*p*-DIOXIN

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
49	2.2	77	11.4	102	1.9	155	44.6
50	14.8	79	9.0	104	.8	156	5.6
51	14.3	80	2.3	107	2.0	157	.6
52	10.1	81	2,8	109	31.8	161	1.8
53	5.1	82	1.3	110	13.9	162	6.9
54	1.0	84	2.3	112	1.0	163	1.5
55	1.1	85	1.6	113	1.2	164	3.0
60	1.1	86	4.0	114	.7	165	.5
61	3.1	87	2.8	115	.5	183	5.3
62	6.5	88	1.3	123	.5	184	.9
63	25.1	89	1.3	125	3.0	189	3.1
64	5.9	90	.5	126	18.5	190	2.3
65	.8	91	1.1	127	39.8	191	1.3
66	.5	92	2.3	128	5.2	192	1.0
68	2.3	93	.7	129	1.6	218	100.0
69	1.5	96	1.4	135	.5	219	14.4
72	1.5	97	1.5	136	6.2	220	33.1
73	3.7	98	2.5	137	1.0	221	4.8
74	14.7	99	2.6	138	3.1	222	.5
75	17.5	100	2.1	139	2.6	253	.5
76	7.2	101	7.4	140	.6		

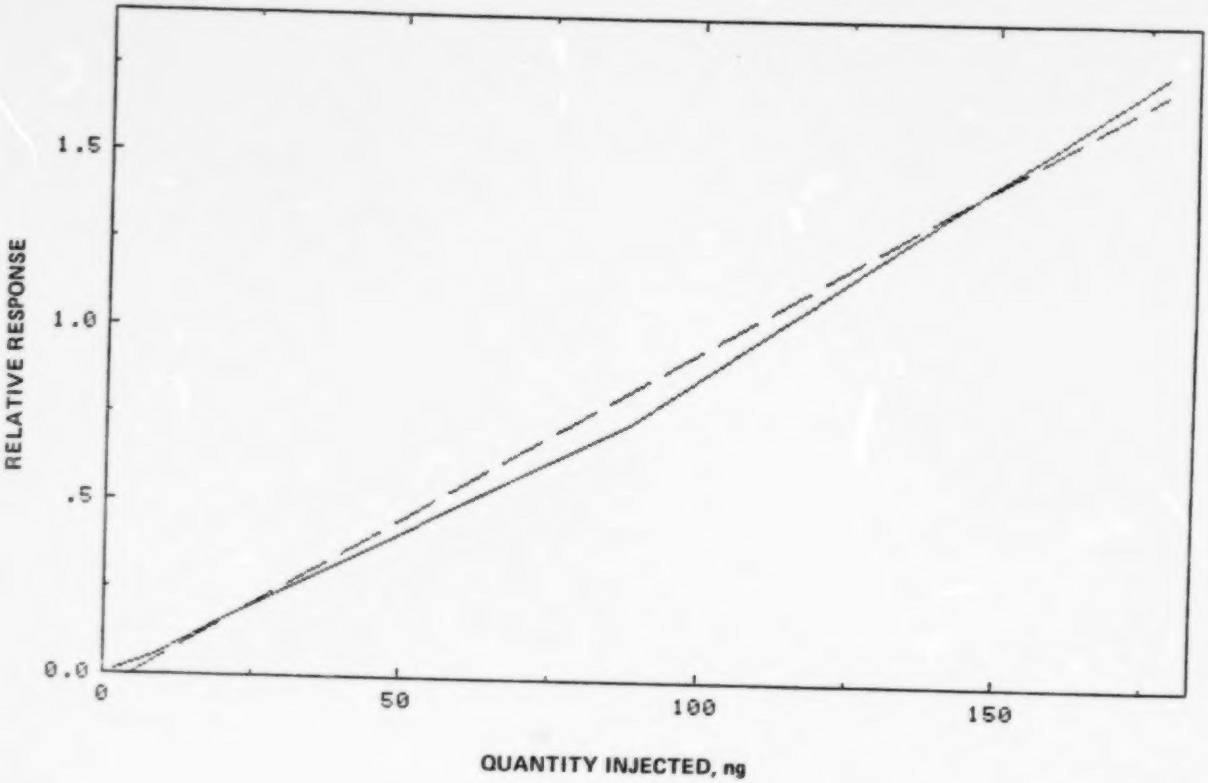
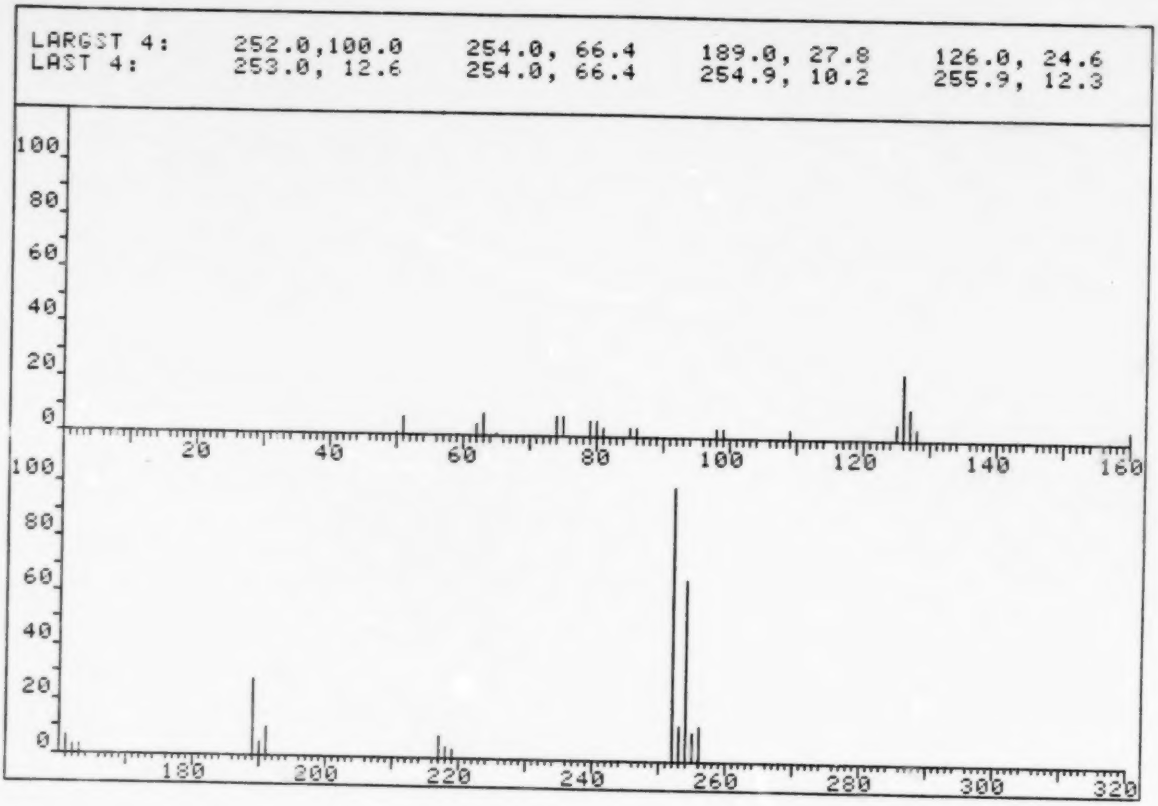


Figure B-18. Calibration curve for the determination of 1-chlorodibenzo-p-dioxin by GC/MS

Range: 9 - 180 ng
Correlation coefficient: 0.997
Slope: 9.8×10^{-3}
y-Intercept: -4.1×10^{-2}



```
LARGST 4:     252.0,100.0     254.0, 66.4     189.0, 27.8     126.0, 24.6
LAST 4:       253.0, 12.6     254.0, 66.4     254.9, 10.2     255.9, 12.3
```

Figure B-19. Mass spectrum for 2,7-dichlorodibenzo-p-dioxin.

TABLE B-9. KEY IONS AND RELATIVE ABUNDANCES FOR 2,7-DICHLORODIBENZO-p-DIOXIN

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
49	.6	85	1.6	114	.8	189	38.6
50	2.9	86	3.2	115	.6	190	5.0
51	3.8	87	1.5	123	.9	191	13.3
53	1.0	88	1.3	124	.6	192	1.7
60	1.0	89	.5	125	6.2	196	.8
61	1.5	94	1.7	126	40.3	198	.5
62	3.6	95	.7	127	15.7	217	9.0
63	8.4	97	1.1	128	3.2	218	1.7
64	.5	98	4.0	135	1.1	219	3.3
72	.7	99	3.7	136	1.0	220	.6
73	2.1	100	1.5	138	1.3	223	.8
74	7.5	101	.5	154	.9	225	.5
75	8.6	107	.6	159	.6	252	100.0
76	1.3	108	1.4	160	5.7	253	13.9
77	1.0	109	1.4	161	8.8	254	63.6
79	7.3	110	2.8	162	2.6	255	9.1
80	4.9	111	1.5	163	3.1	256	11.0
81	1.5	112	1.0	170	.5	257	1.4
84	1.5	113	1.5	173	.7		

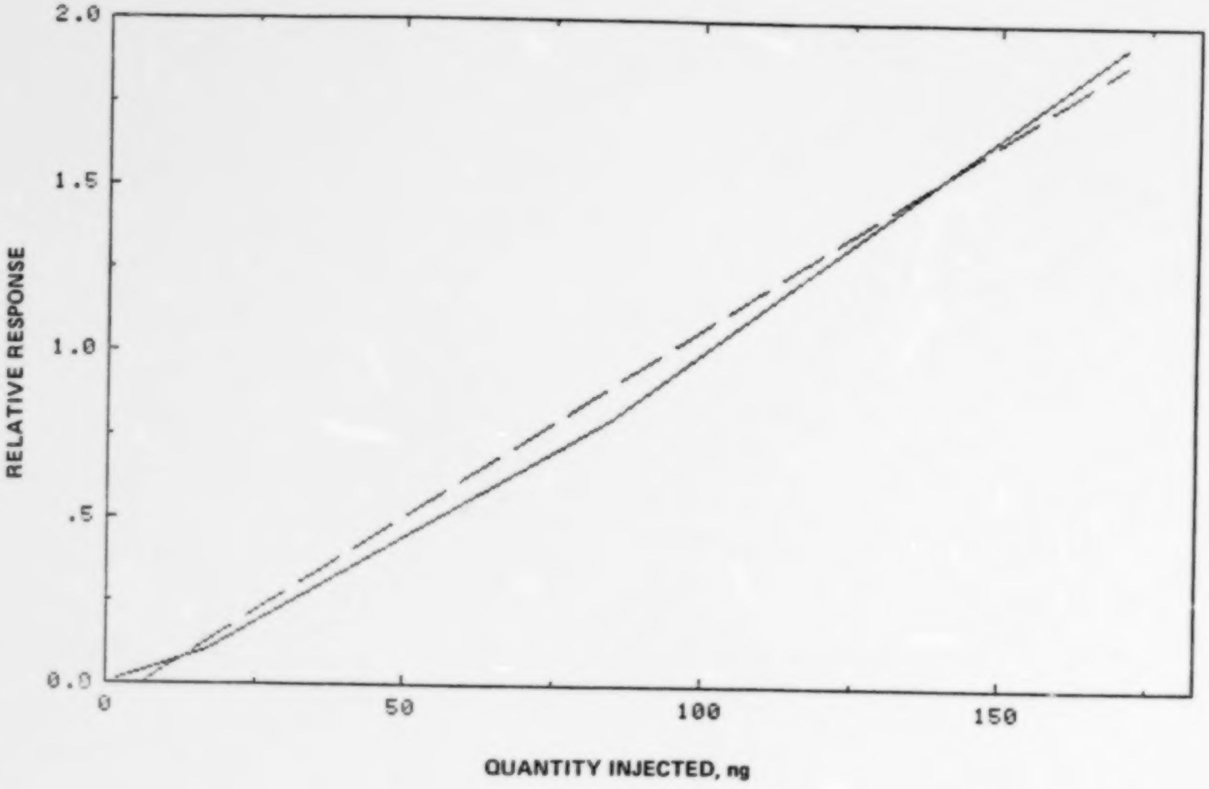
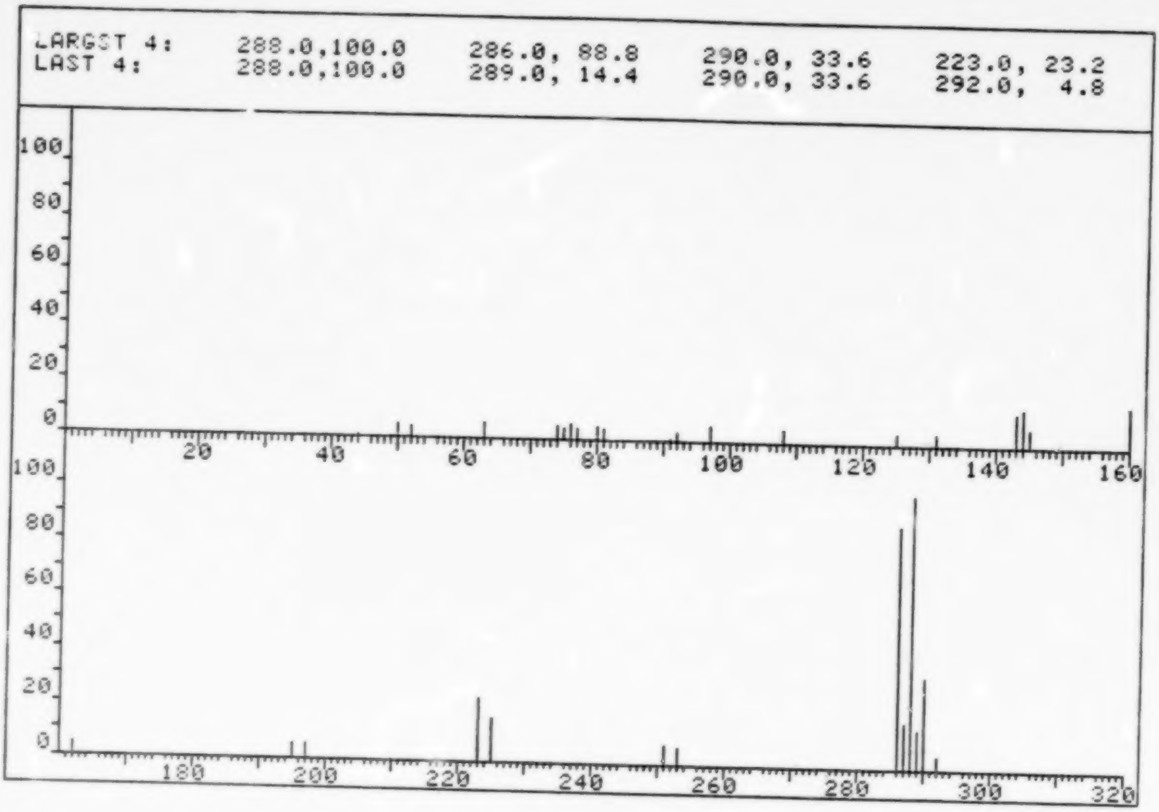


Figure B-20. Calibration curve for the determination of 2,7-dichlorodibenzo-p-dioxin by GC/MS.

Range: 9 - 170 ng
Correlation coefficient: 0.997
Slope: 1.1×10^{-2}
y-Intercept: -6.4×10^{-2}



```
LARGST 4:      288.0,100.0     286.0, 88.8     290.0, 33.6     223.0, 23.2
LAST 4:        288.0,100.0     289.0, 14.4     290.0, 33.6     292.0,  4.8
```

Figure B-21. Mass spectrum for 1,2,4-trichlorodibenzo-p-dioxin.

TABLE B-10. KEY IONS AND RELATIVE ABUNDANCES FOR 1,2,4-TRICHLORODIBENZO-*p*-DIOXIN

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
47	.9	99	6.4	149	3.5	205	.6
49	4.0	100	.8	151	.6	206	2.6
50	17.8	101	.8	153	3.4	207	2.2
51	10.0	102	2.0	154	2.4	208	1.2
52	14.2	103	2.5	155	.5	209	1.3
53	5.1	104	2.7	156	2.2	216	2.8
54	1.3	106	.9	157	.7	217	.6
55	.7	107	3.5	158	1.4	218	1.0
60	2.0	108	18.1	159	5.2	223	42.6
61	6.6	109	5.0	160	44.8	224	6.1
62	5.7	110	4.7	161	6.4	225	27.2
63	13.1	111	5.7	162	13.3	226	3.5
64	7.1	113	2.5	163	1.7	227	4.1
65	1.1	115	1.8	166	.7	228	.6
66	.6	116	1.8	168	1.1	229	.5
67	.8	117	.7	169	.9	230	3.1
68	.6	118	2.7	170	.9	231	1.1
71	1.8	119	8.4	171	.7	232	3.3
72	2.0	120	2.6	172	.8	233	.6
73	6.7	121	6.7	175	1.8	234	1.3
74	13.5	122	1.5	177	1.3	251	8.8
75	14.6	123	4.8	178	1.7	252	1.3
76	9.9	124	4.1	179	.6	253	5.9
77	6.5	125	11.3	180	1.4	254	1.1
78	.8	126	2.1	181	1.0	255	1.1
79	3.4	130	2.0	182	.8	257	2.0
80	12.5	131	7.5	184	.6	258	2.0
81	3.4	132	1.3	187	1.4	259	1.7
83	2.0	133	5.9	188	1.7	260	1.7
84	10.8	134	1.0	189	.7	261	.8
85	3.4	135	2.1	190	.6	262	.8
86	5.0	136	.8	191	.7	286	96.3
87	7.0	137	1.0	193	.6	287	16.6
89	2.4	139	.6	194	4.4	288	100.0
92	2.6	143	35.1	195	11.9	289	14.2
94	1.8	144	31.9	196	4.1	290	31.2
95	2.7	145	13.2	197	7.7	291	3.8
96	9.0	146	2.0	198	1.6	292	3.1
97	10.2	147	5.9	199	1.6		
98	11.0	148	.5	204	2.5		

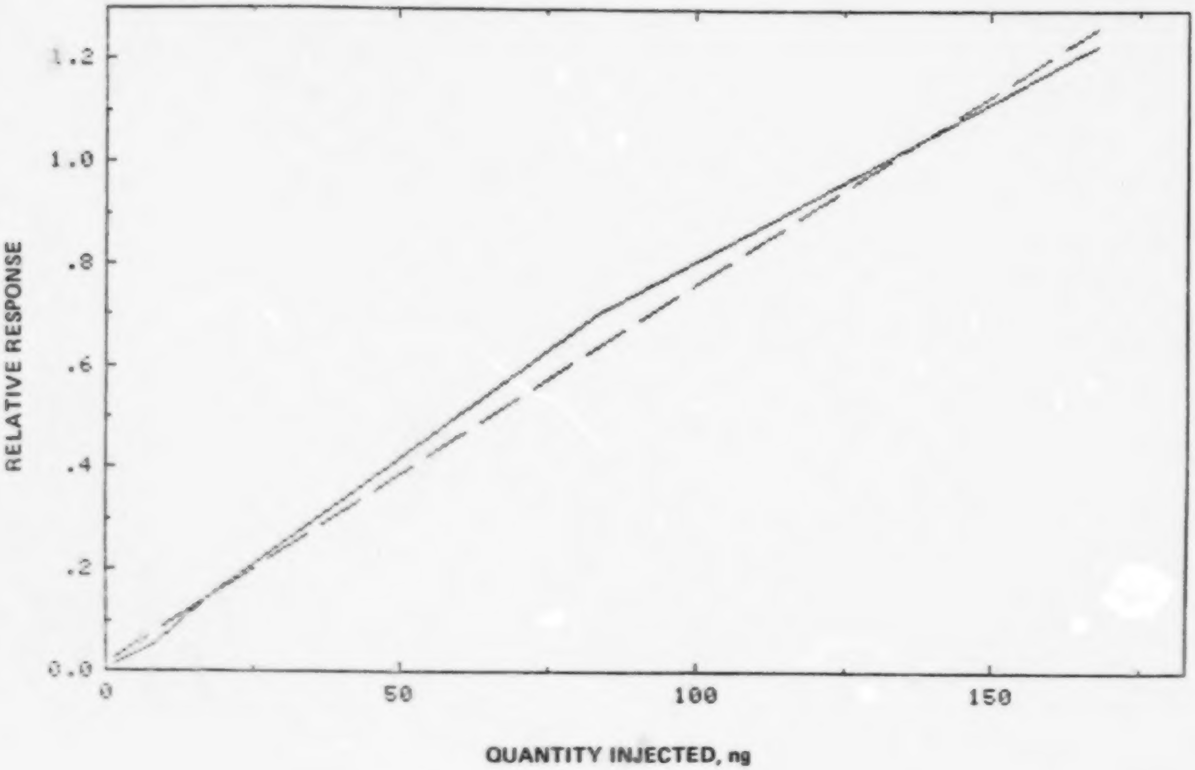
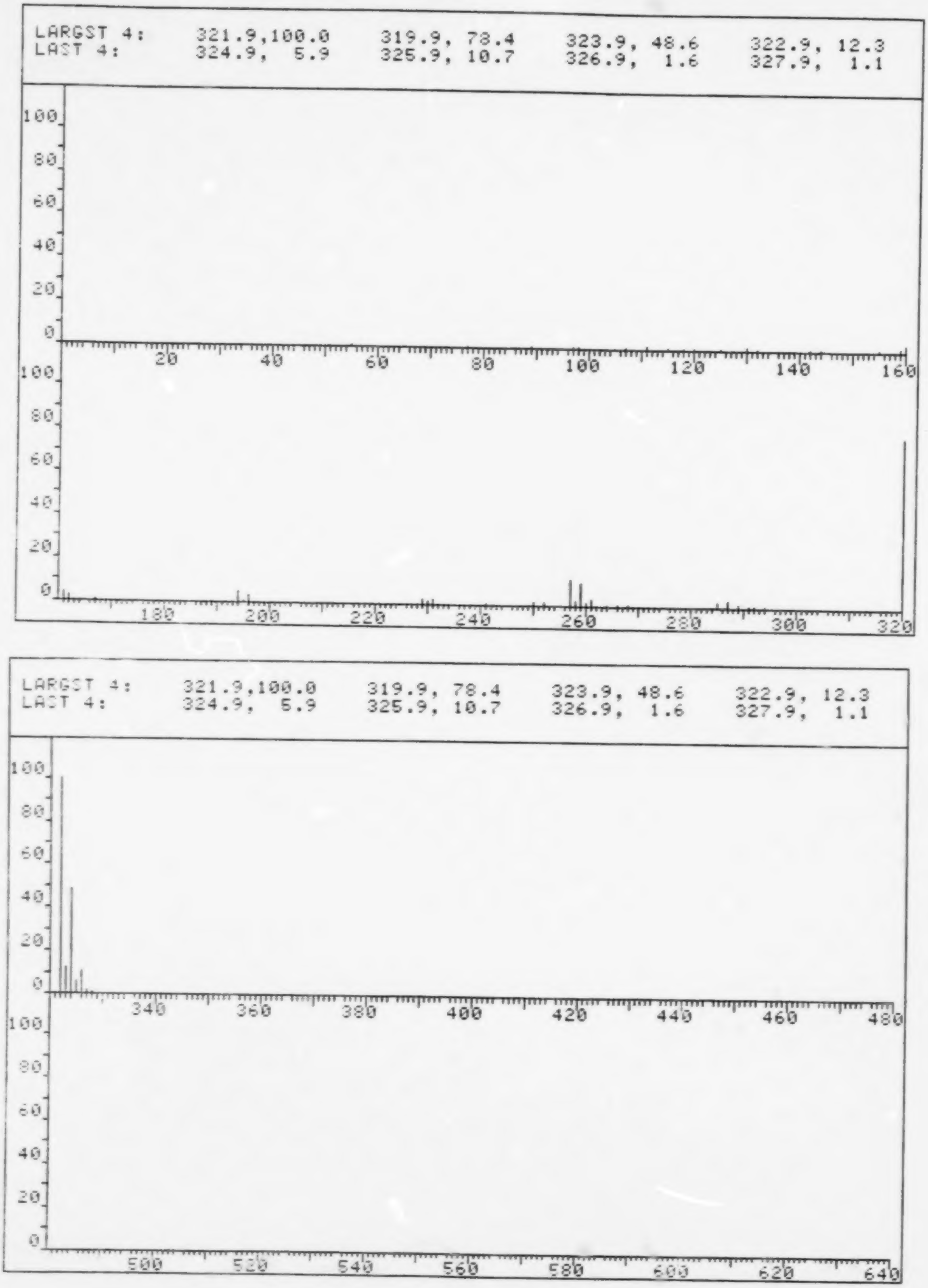


Figure B-22. Calibration curve for the determination of 1,2,4-trichlorodibenzo-p-dioxin by GC/MS.

Range: 10 - 170 ng
Correlation coefficient: 0.999
Slope: 7.4×10^{-3}
y-Intercept: 1.4×10^{-2}



```
LARGST 4:     321.9,100.0     319.9, 78.4     323.9, 48.6     322.9, 12.3
LAST 4:       324.9,  5.9     325.9, 10.7     326.9,  1.6     327.9,  1.1
```

```
LARGST 4:     321.9,100.0     319.9, 78.4     323.9, 48.6     322.9, 12.3
LAST 4:       324.9,  5.9     325.9, 10.7     326.9,  1.6     327.9,  1.1
```

Figure B-23. Mass spectrum for 1,2,3,4-tetrachlorodibenzo-p-dioxin.

TABLE B-11. KEY IONS AND RELATIVE ABUNDANCES FOR 1,2,3,4-TETRACHLORO-DIBENZO-*p*-DIOXIN

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
50	1.7	115	.8	196	3.3	287	3.3
51	.9	118	.8	229	2.1	289	1.4
52	1.0	120	1.1	230	1.0	291	.9
55	.9	125	.9	231	2.3	292	.8
63	1.5	130	1.3	241	.8	294	.9
64	.8	132	.8	250	1.3	320	78.3
74	1.0	142	1.2	252	1.1	322	100.0
75	1.3	144	1.0	257	11.6	323	12.3
76	1.0	155	.9	258	2.0	324	48.6
77	1.0	159	1.3	259	9.9	325	5.9
89	.8	160	3.9	260	1.4	326	10.7
95	.9	161	5.3	261	3.0	327	1.6
97	1.7	162	1.8	264	.8	328	1.1
98	1.4	167	.8	266	.8		
107	.9	194	4.9	268	.9		
111	1.3	195	.9	285	2.4		

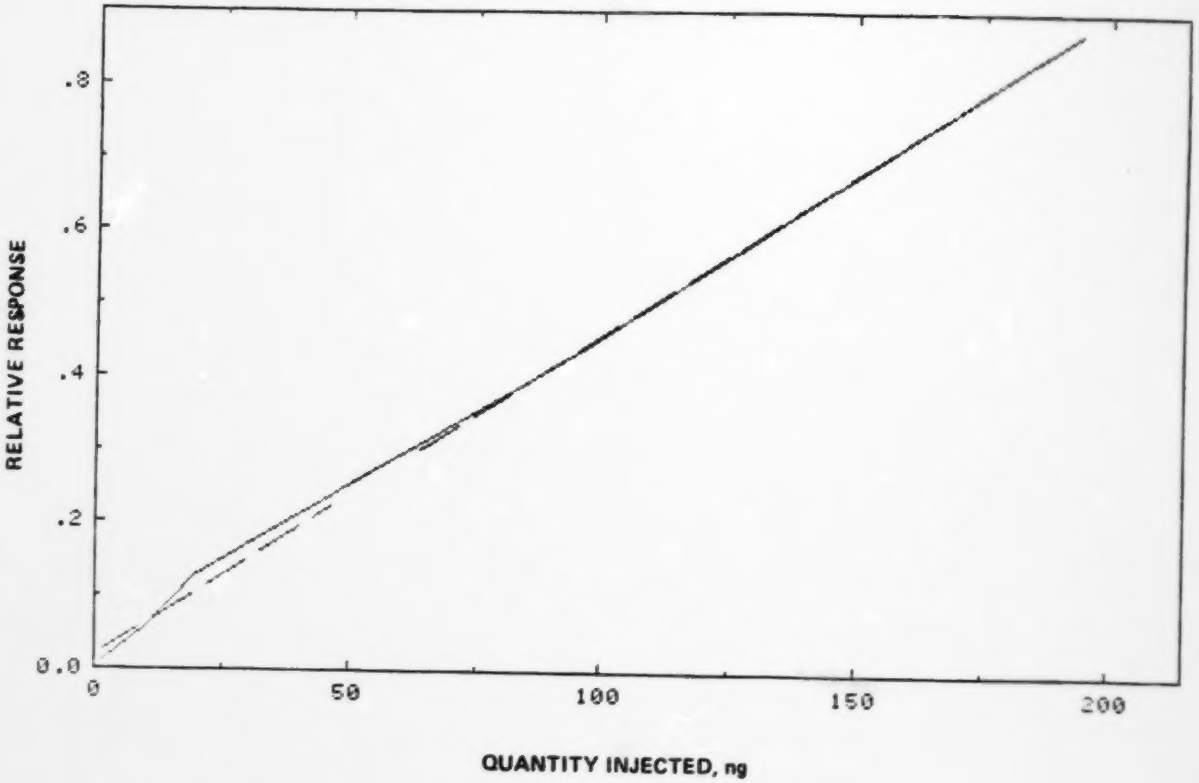
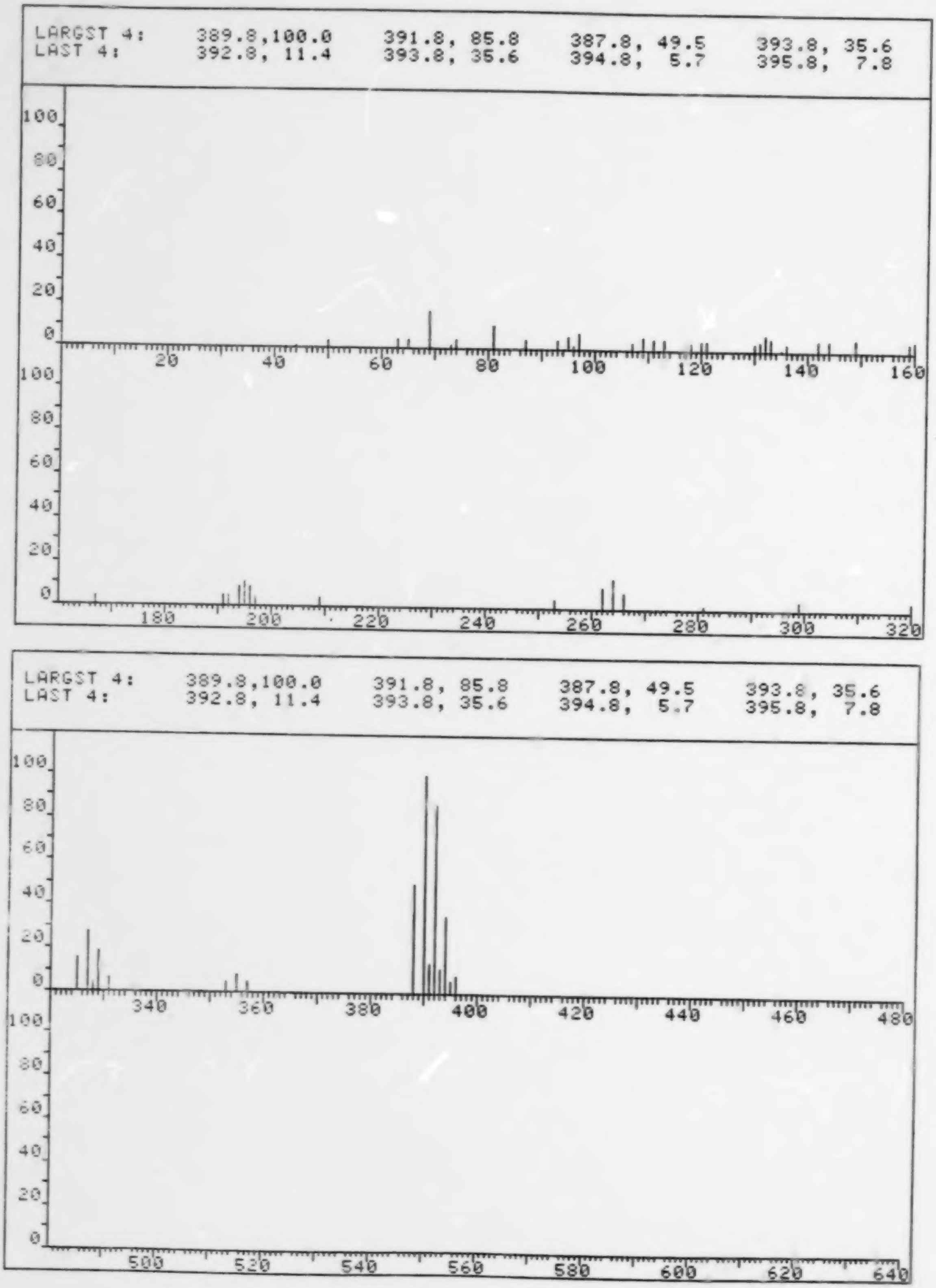


Figure B-24. Calibration curve for the determination of 1,2,3,4-tetrachlorodibenzo-p-dioxin by GC/MS.

Range: 10 - 190 ng
Correlation coefficient: 0.999
Slope: 4.4 x 10^{-}[illegible]
y-Intercept: 1.9 x 10^{-2}



```
LARGST 4:     389.8,100.0     391.8, 85.8     387.8, 49.5     393.8, 35.6
LAST 4:       392.8, 11.4     393.8, 35.6     394.8,  5.7     395.8,  7.8
```

```
LARGST 4:     389.8,100.0     391.8, 85.8     387.8, 49.5     393.8, 35.6
LAST 4:       392.8, 11.4     393.8, 35.6     394.8,  5.7     395.8,  7.8
```

Figure B-25. Mass spectrum for 1,2,3,4,7,8-hexachlorodibenzo-p-dioxin.

TABLE B-12. KEY IONS AND RELATIVE ABUNDANCES FOR 1,2,3,4,7,8-HEXACHLORO-DIBENZO-p-DIOXIN

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
47	1.9	113	19.0	168	3.5	283	1.6
49	1.9	115	5.8	169	3.6	289	1.2
50	6.7	118	10.0	177	6.0	290	3.0
51	1.4	119	2.3	178	2.8	291	1.1
53	1.7	120	12.9	179	5.5	292	3.5
60	3.8	121	3.1	181	6.0	294	1.5
61	5.3	122	6.8	183	5.3	296	1.2
62	7.2	123	1.2	185	1.9	297	3.6
63	8.1	127	1.4	188	2.7	298	1.5
65	2.4	130	12.4	190	2.6	299	5.6
71	3.0	131	20.7	191	3.0	300	1.7
72	3.2	132	30.1	192	12.6	301	3.3
73	6.1	133	11.9	194	26.8	303	1.1
74	17.7	134	5.9	195	41.9	311	1.1
75	1.7	135	1.7	196	30.4	318	4.3
76	1.1	136	1.6	197	13.0	320	4.7
77	3.2	137	2.0	198	3.3	322	2.5
78	1.8	138	2.0	200	1.1	325	28.3
82	1.1	142	20.6	202	1.4	327	50.4
83	4.2	143	4.9	209	2.2	328	5.9
84	6.6	144	19.0	211	1.7	329	30.5
85	10.2	145	4.1	212	1.9	330	4.2
86	2.6	146	6.3	214	3.2	331	10.2
87	10.6	147	2.5	216	1.6	332	1.8
88	1.4	148	4.4	218	1.2	333	1.7
89	2.8	149	3.5	226	2.3	353	6.4
90	1.2	150	4.2	227	6.5	355	10.4
94	2.7	151	1.9	228	3.2	356	1.6
95	9.3	153	7.2	229	7.3	357	6.5
96	6.6	155	8.3	230	1.8	359	2.0
97	21.7	156	3.8	231	2.4	388	57.9
98	3.6	157	5.7	255	5.6	390	100.0
99	5.3	158	2.3	256	1.2	391	16.0
102	1.1	159	10.0	257	5.3	392	89.8
106	2.6	160	13.7	259	1.5	393	11.6
107	8.8	161	6.7	262	25.8	394	39.0
108	9.6	162	8.0	264	37.9	395	4.6
109	18.3	163	11.3	265	5.1	396	9.4
110	4.6	164	11.1	266	18.3	397	1.6
111	13.3	166	2.8	267	2.5	398	1.2
112	1.5	167	10.5	268	4.2		

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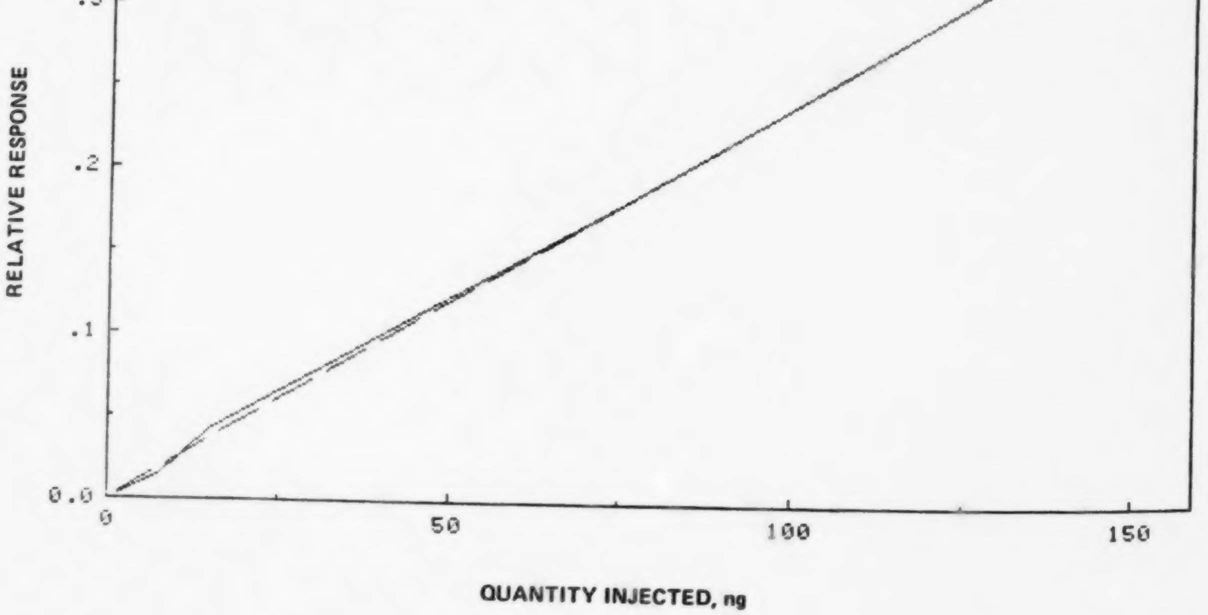


Figure B-26. Calibration curve for the determination of 1,2,3,4,7,8-hexachlorodibenzo-*p*-dioxin by GC/MS.

Range: 8 - 150 ng
Correlation coefficient: 0.999
Slope: 2.4×10^{-3}
y-Intercept: 3.8×10^{-4}

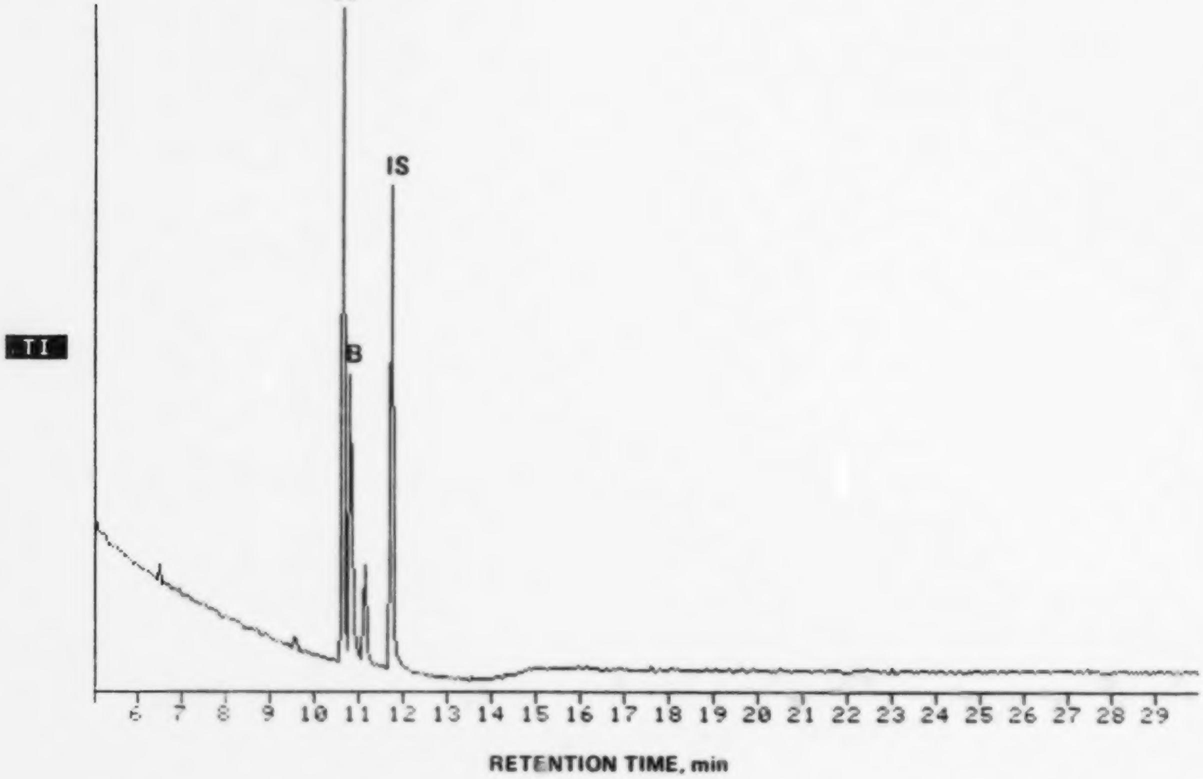
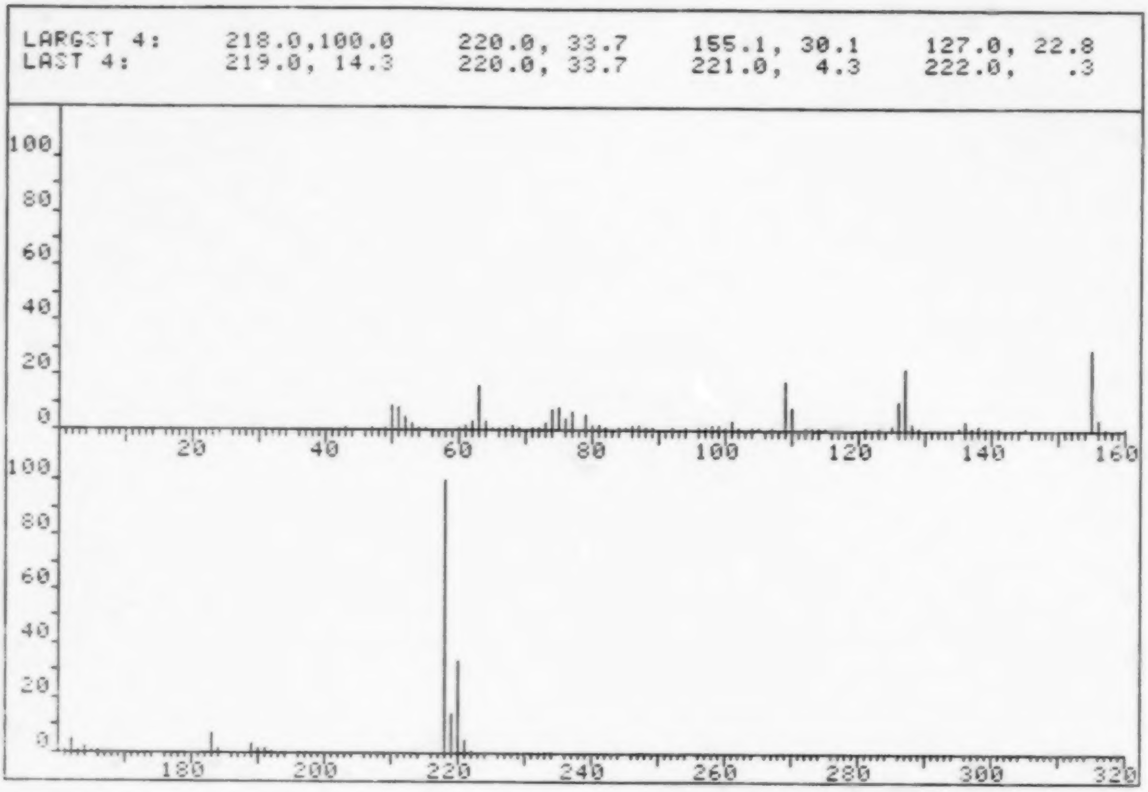


Figure B-27. Chromatogram by GC/MS for

(A) 2-chlorodibenzo-p-dioxin(t_R = 10.6 min) and
(B) 2,3-dichlorodibenzo-p-dioxin(t_R = 10.8 min).

Temperature program:
60 °C, 3 min isothermal
60 to 300 °C at 20 °C/min
300 °C, 10 min isothermal



```
LARGST 4:    218.0,100.0    220.0, 33.7    155.1, 30.1    127.0, 22.8
LAST 4:      219.0, 14.3    220.0, 33.7    221.0,  4.3    222.0,    .3
```

Figure B-28. Mass spectrum for 2-chlorodibenzo-p-dioxin.

TABLE B-13. KEY IONS AND RELATIVE ABUNDANCES FOR 2-CHLORODIBENZO-p-DIOXIN

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
49	.9	76	4.1	102	1.1	156	4.0
50	8.6	77	6.6	104	.7	161	1.0
51	8.6	79	5.9	107	.6	162	4.6
52	5.0	80	1.4	109	18.2	163	.8
53	2.2	81	1.9	110	7.6	164	1.8
54	.5	82	.6	112	.5	183	7.0
60	1.0	84	.6	113	.7	184	1.0
61	1.6	85	.6	125	1.5	189	2.5
62	3.8	86	1.8	126	10.7	190	1.2
63	16.0	87	1.4	127	22.8	191	1.0
64	3.7	88	1.0	128	2.8	218	100.0
68	1.7	96	1.1	129	.6	219	14.3
69	.7	97	.8	136	2.9	220	33.7
72	.6	98	1.6	137	.5	221	4.3
73	2.1	99	1.3	138	1.5		
74	7.5	100	1.1	139	1.2		
75	6.4	101	3.6	155	30.1		

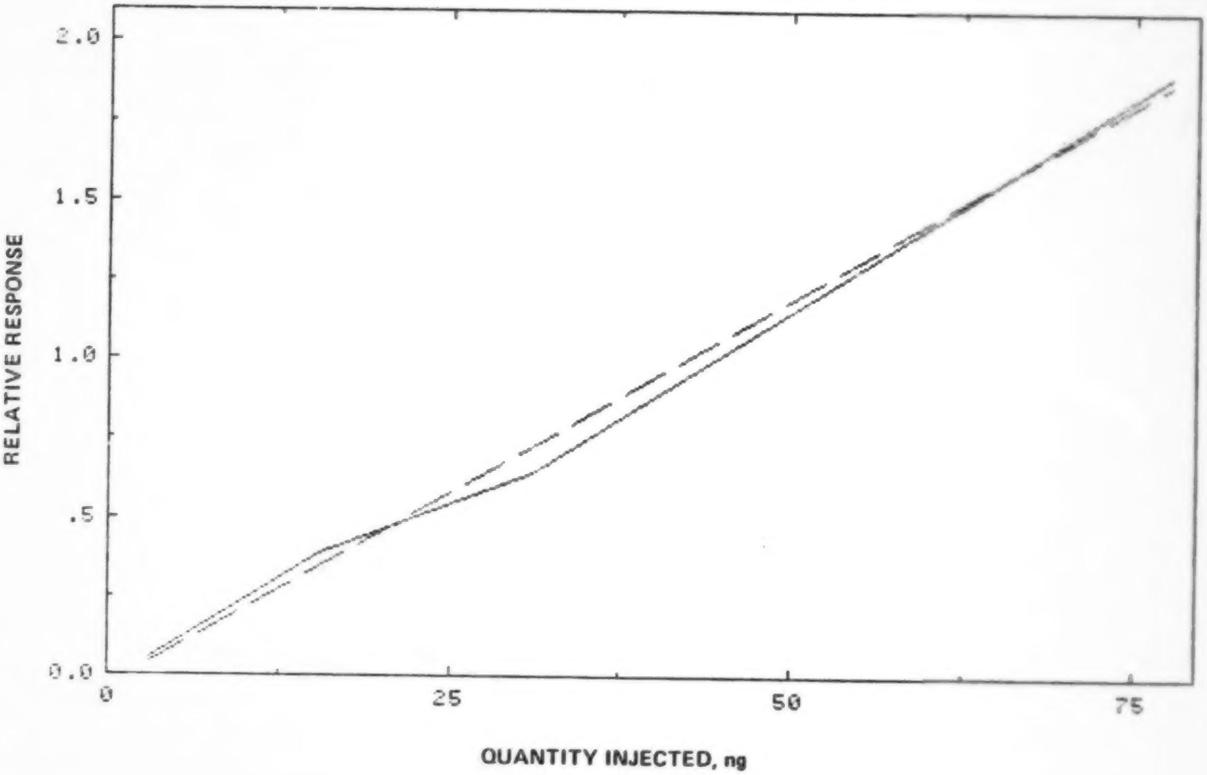


Figure B-29. Calibration curve for the determination of 2-chlorodibenzo-p-dioxin by GC/MS.

Range: 10 - 80 ng
Correlation coefficient: 0.997
Slope: 2.5×10^{-2}
y-Intercept: -3.8×10^{-2}

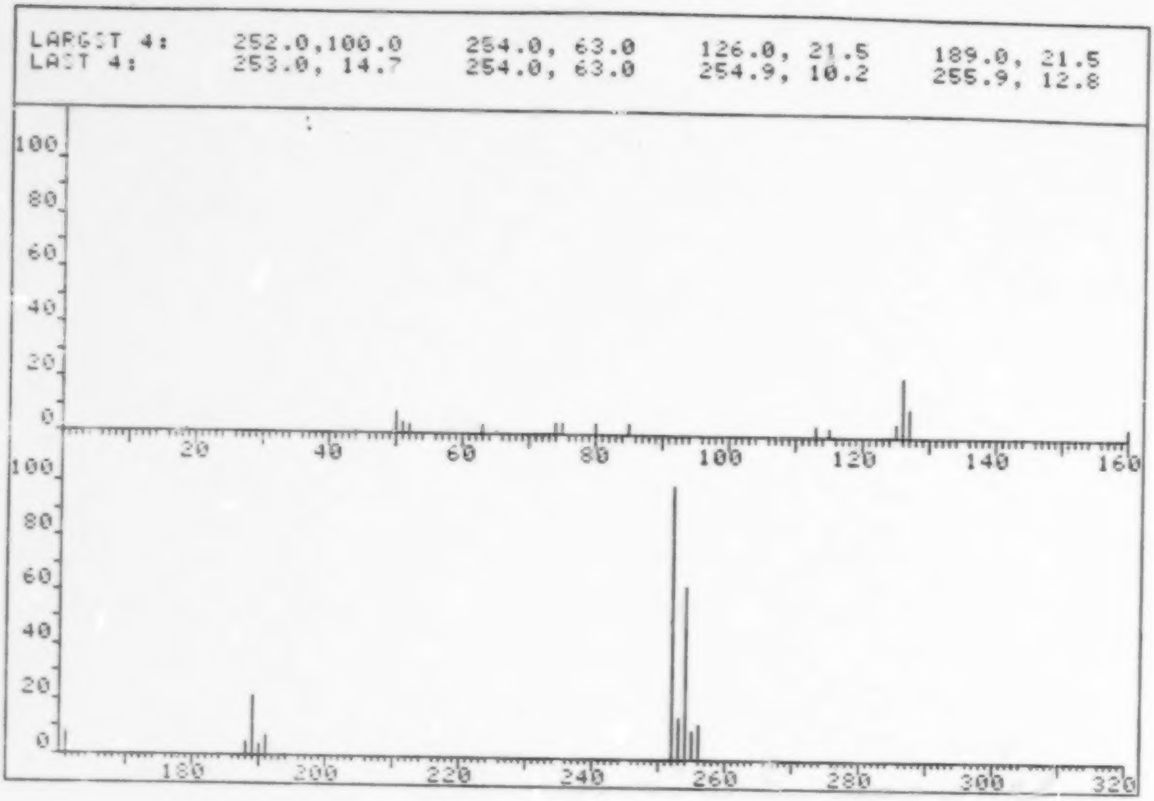


Figure B-30. Mass spectrum for 2,3-dichlorodibenzo-p-dioxin.

TABLE B-14. KEY IONS AND RELATIVE ABUNDANCES FOR 2,3-DICHLORODIBENZO-*p*-DIOXIN

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
49	.6	87	1.5	125	4.8	189	30.1
50	4.3	91	.7	126	31.3	190	3.8
51	1.9	92	.8	127	14.4	191	9.8
52	2.0	94	1.3	128	2.4	192	1.3
53	1.2	95	.8	132	.6	195	.6
60	.6	97	4.0	133	.6	196	2.2
61	1.3	98	2.0	134	.8	197	.6
62	2.3	99	3.2	135	.6	198	1.9
63	4.4	100	.8	136	.6	217	5.0
64	1.4	104	.7	144	.7	218	.8
72	.6	107	.6	146	.6	219	1.6
73	1.3	108	1.1	153	1.0	223	2.1
74	5.4	109	2.2	154	.7	224	1.0
75	3.4	110	.5	159	.6	225	1.5
76	2.8	111	1.0	160	4.9	226	.8
77	1.3	113	4.7	161	10.6	252	100.0
79	.6	114	1.1	162	2.8	253	14.1
80	4.1	115	1.4	163	3.0	254	65.0
81	1.4	120	.8	170	1.5	255	9.3
84	1.3	122	.7	172	1.1	256	11.4
85	3.0	123	.7	173	.8	257	1.2
86	1.7	124	.6	182	2.0		

Reproduced from best available copy.

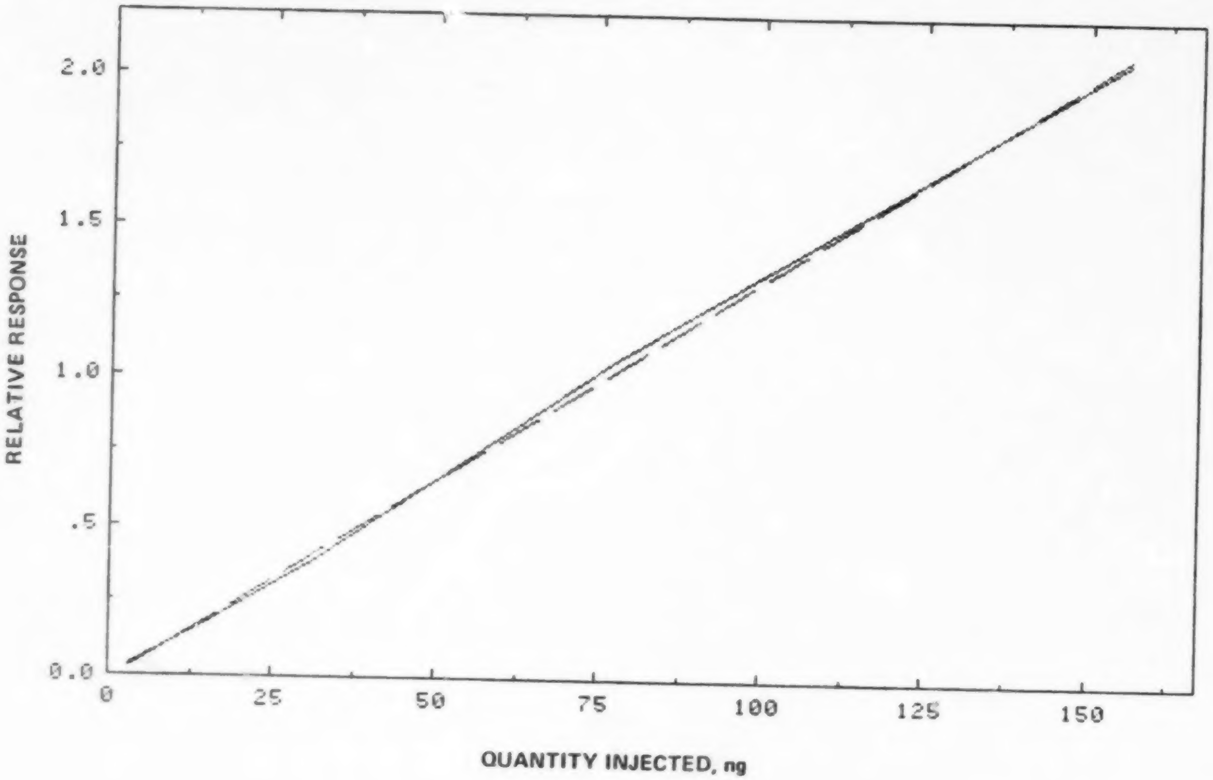


Figure B-31. Calibration curve for the determination of 2,3-dichlorodibenzo-p-dioxin by GC/MS.

Range: 3.0 - 160 ng
Correlation coefficient: 0.999
Slope: 1.3×10^{-2}
y-Intercept: -1.3×10^{-2}

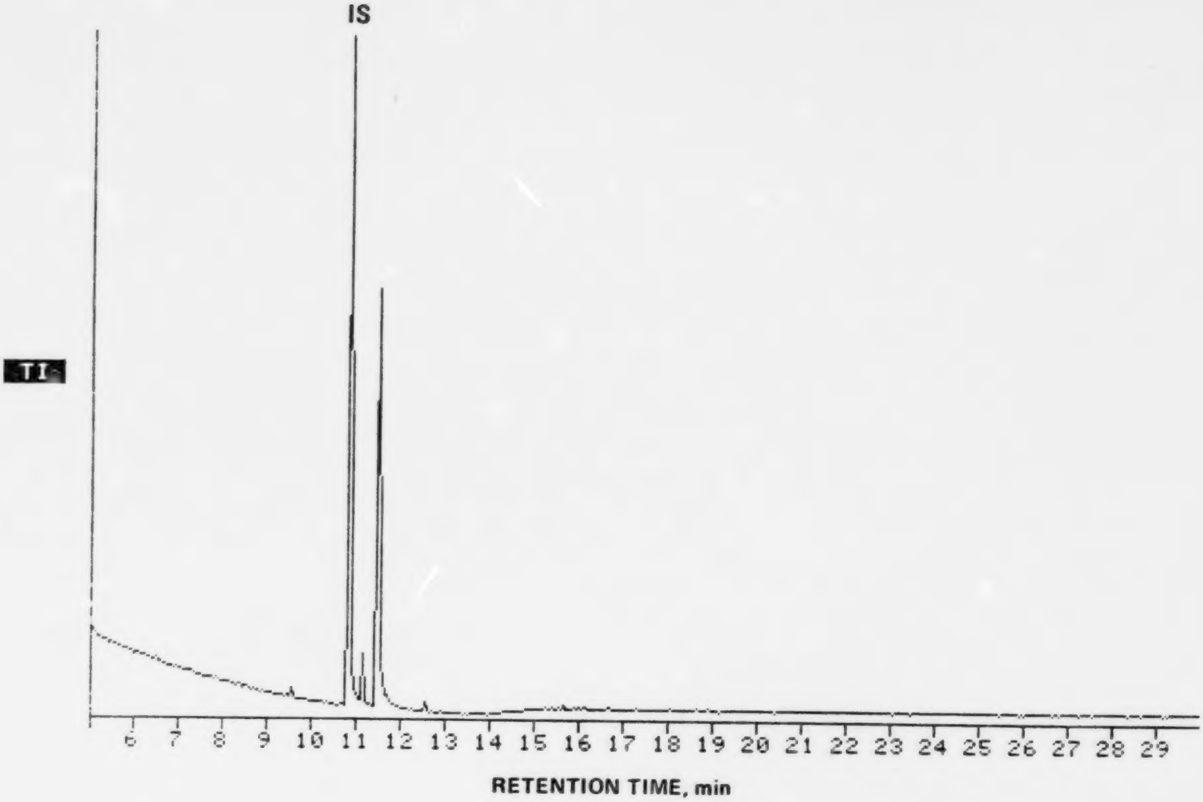
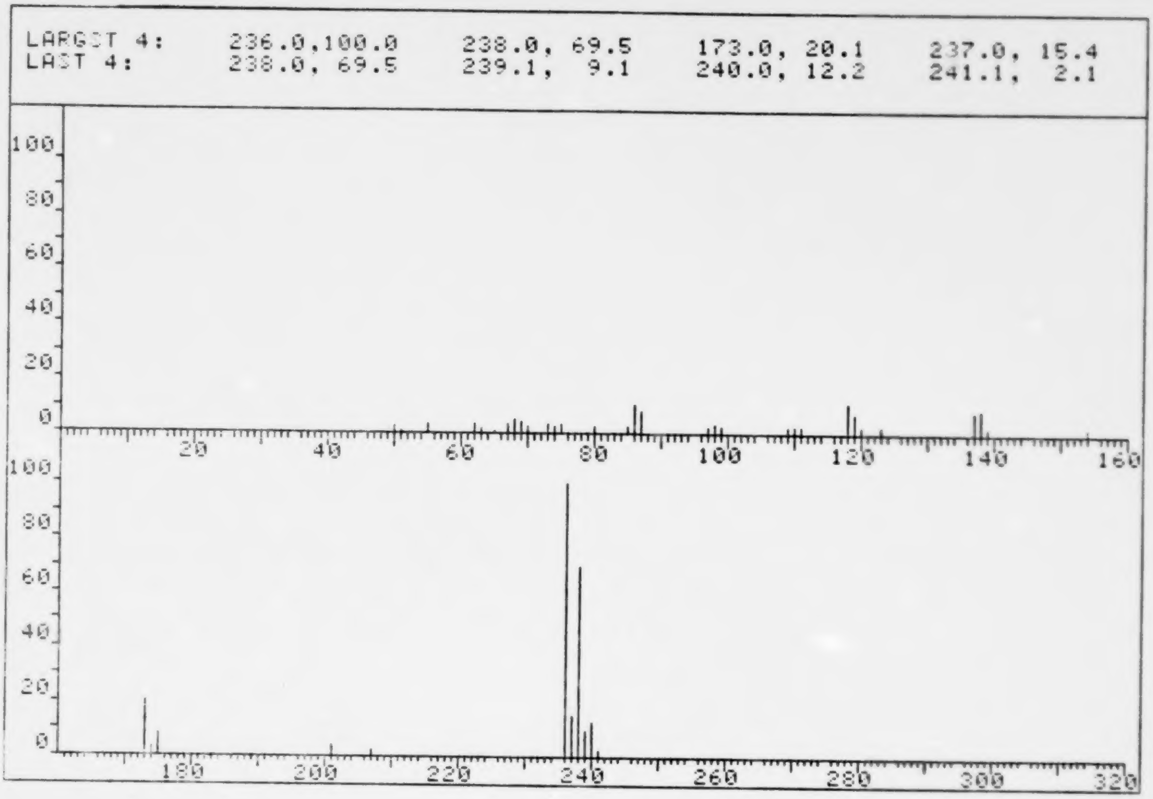


Figure B-32. Chromatogram by GC/MS for 3,6-dichlorodibenzofuran (t_R = 11.5 min).

Temperature program:
60 °C, 3 min isothermal
60 to 300 °C at 20 °C/min
300 °C, 10 min isothermal



```
LARGST 4:      236.0,100.0     238.0, 69.5     173.0, 20.1     237.0, 15.4
LAST 4:        238.0, 69.5     239.1,  9.1     240.0, 12.2     241.1,  2.1
```

Figure B-33. Mass spectrum for 3,6-dichlorodibenzofuran.

TABLE B-15. KEY IONS AND RELATIVE ABUNDANCES FOR 3,6-DICHLORODIBENZOFURAN

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
62	.6	110	.6	173	12.4	209	.6
68	1.5	111	.6	174	1.8	236	100.0
69	.9	118	3.9	175	4.5	237	13.9
73	.7	119	2.9	176	.6	238	67.9
74	.7	120	.7	200	.6	239	8.8
86	2.8	137	3.1	201	1.8	240	11.8
98	.6	138	3.2	203	.5	241	1.4
99	.6	147	.8	207	1.1		

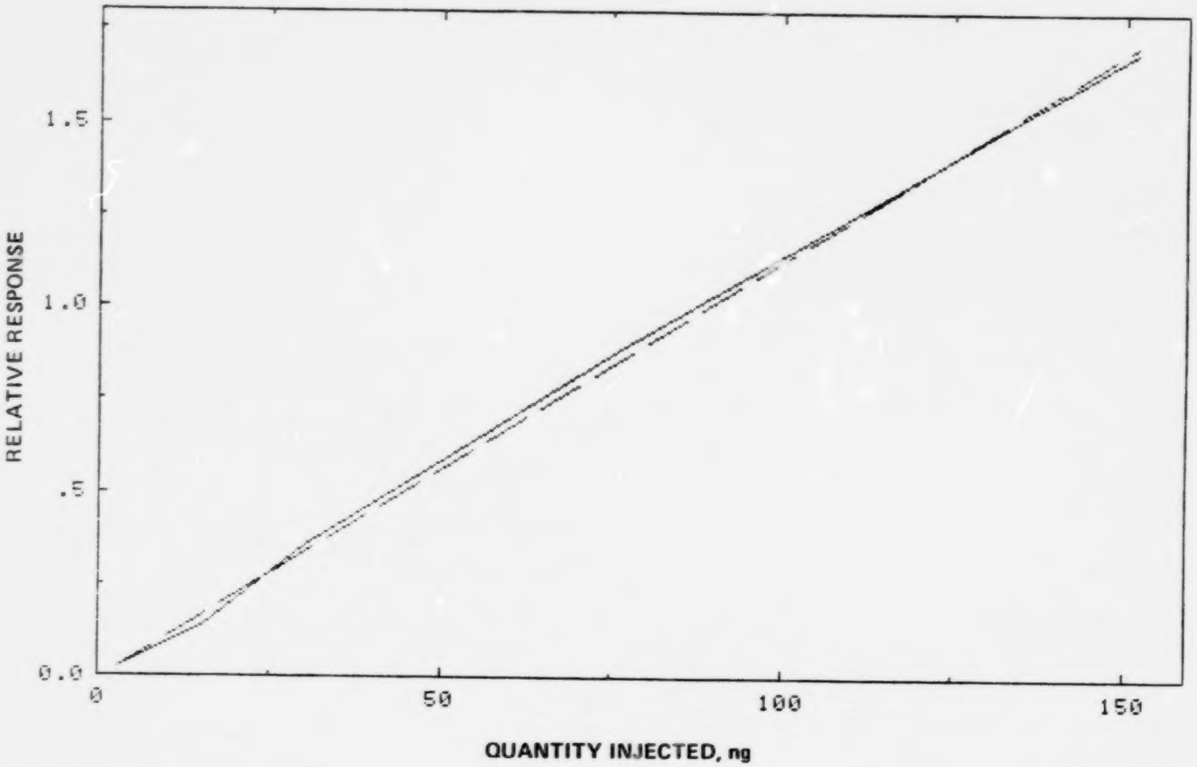


Figure B-34. Calibration curve for the determination of 3,6-dichlorodibenzofuran by GC/MS.

Range: 3.0 - 150 ng
Correlation coefficient: 0.999
Slope: 1.1×10^{-2}
y-Intercept: -9.0×10^{-3}

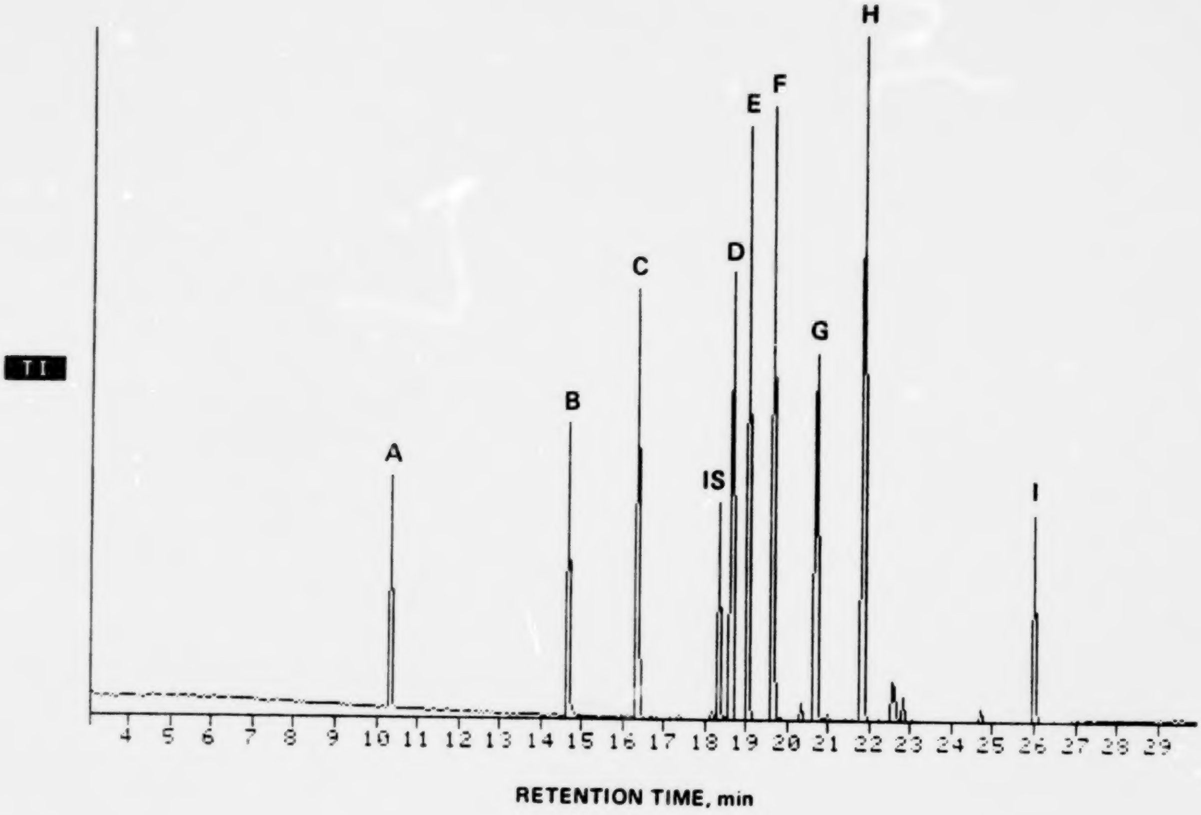


Figure B-35. Chromatogram by GC/MS for

(A) 1-fluoronaphthalene(t_R = 10.4 min),
(B) 3-bromoquinoline(t_R = 14.7 min),
(C) 1-bromo-4-methylnaphthalene(t_R = 16.4 min),
(D) 5-bromoacenaphthene(t_R = 18.7 min),
(E) 1-chloro-8-nitronaphthalene(t_R = 19.1 min),
(F) 2-bromofluorene(t_R = 19.7 min),
(G) 9-cloroanthracene(t_R = 20.7 min),
(H) 9-bromoanthracene(t_R = 21.8 min), and
(I) 3,6-dibromocarbazole(t_R = 26.0 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 1 min isothermal

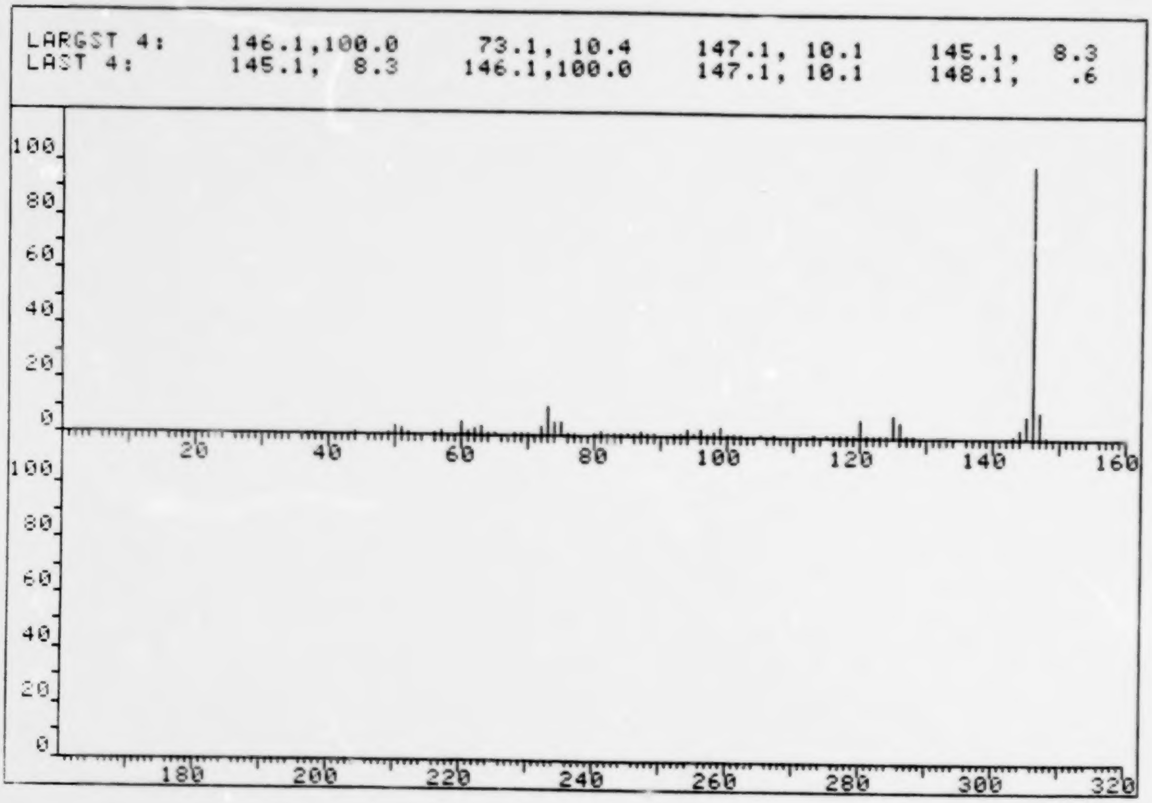


Figure B-36. Mass spectrum for 1-fluoronaphthalene.

TABLE B-16. KEY IONS AND RELATIVE ABUNDANCES FOR 1-FLUORONAPHTHALENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	.1	72	3.4	93	1.1	119	.4
49	.5	73	10.4	94	2.2	120	6.3
50	3.4	74	5.0	95	.9	121	.7
51	2.2	75	4.8	96	2.4	122	.4
52	.3	76	1.2	97	.4	123	.3
56	.3	77	.6	98	1.5	124	.3
57	1.5	80	.3	99	3.0	125	7.7
60	5.3	81	1.5	100	1.1	126	5.6
61	1.4	82	.2	101	.4	127	1.1
62	2.4	83	.2	102	.4	128	.5
63	3.8	84	.2	105	.5	143	.3
64	.3	85	.7	107	.6	144	3.8
64	.3	86	1.2	113	.3	145	8.3
65	.2	87	1.3	115	1.1	146	100.0
68	.8	88	.3	116	.2	147	10.1
69	.7	89	.3	117	.2	148	.6
70	1.1	92	.7	118	.5		

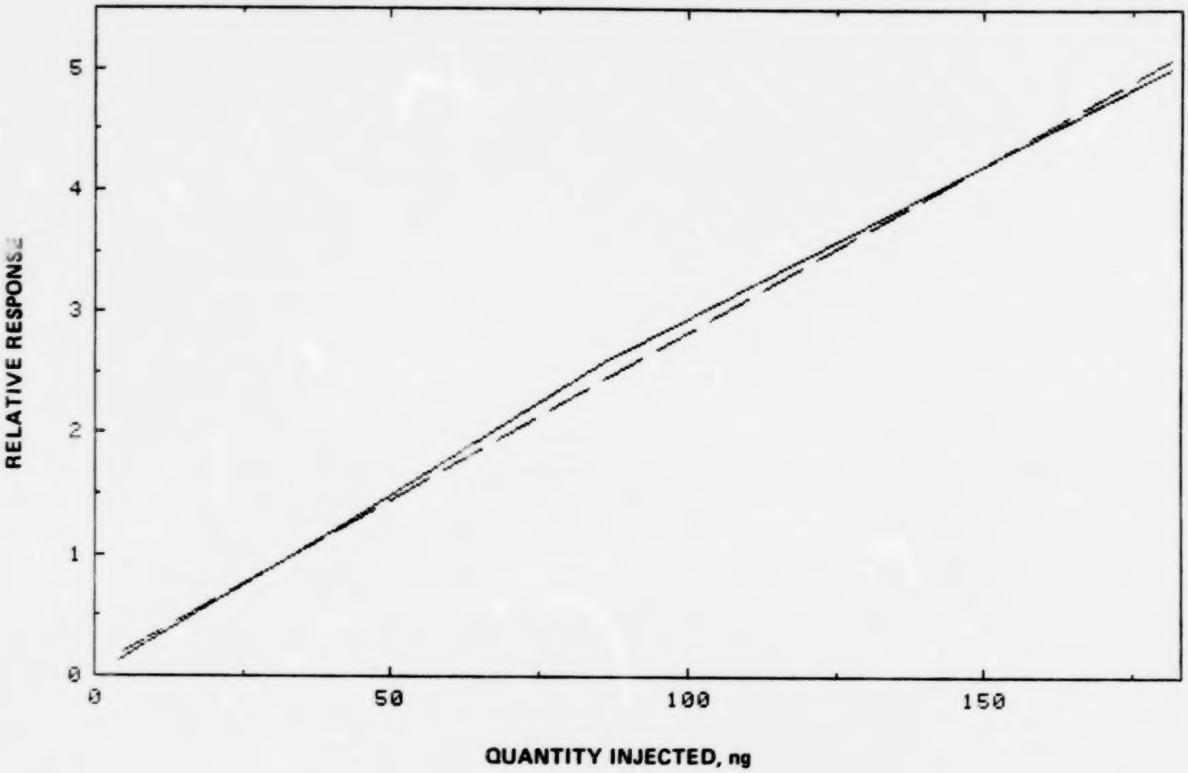
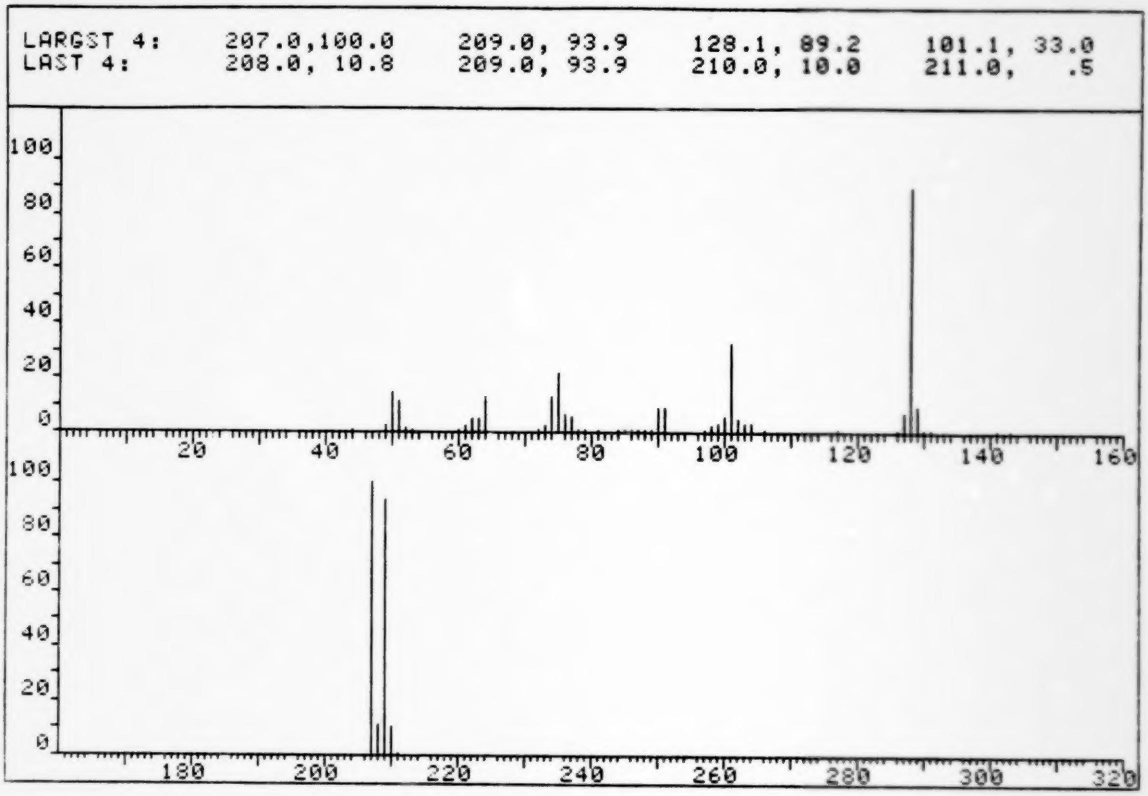


Figure B-37. Calibration curve for the determination of 1-fluoronaphthalene by GC/MS.

Range: 9.0 - 180 ng
Correlation coefficient: 0.999
Slope: 2.8×10^{-2}
y-Intercept: 6.3×10^{-2}



```
LARGST 4:      207.0,100.0     209.0, 93.9     128.1, 89.2     101.1, 33.0
LAST 4:        208.0, 10.8     209.0, 93.9     210.0, 10.0     211.0,    .5
```

Figure B-38. Mass spectrum for 3-bromoquinoline.

TABLE B-17. KEY IONS AND RELATIVE ABUNDANCES FOR 3-BROMOQUINOLINE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	.2	74	12.4	90	9.1	127	7.4
49	2.3	75	22.1	91	9.1	128	89.2
50	14.8	76	6.2	97	.5	129	9.7
51	11.5	77	5.8	98	2.5	130	.7
52	2.0	78	1.0	99	3.0	131	.3
53	.3	79	.7	100	5.8	141	.3
60	.4	81	.8	101	33.0	143	.3
61	2.8	84	.1	102	4.8	207	100.0
62	5.0	85	.7	103	3.0	208	10.8
63	5.3	86	1.1	104	3.0	209	93.9
64	12.6	87	1.3	106	.3	210	10.0
72	.3	88	.9	117	.4	211	.5
73	2.1	89	.6	126	.6		

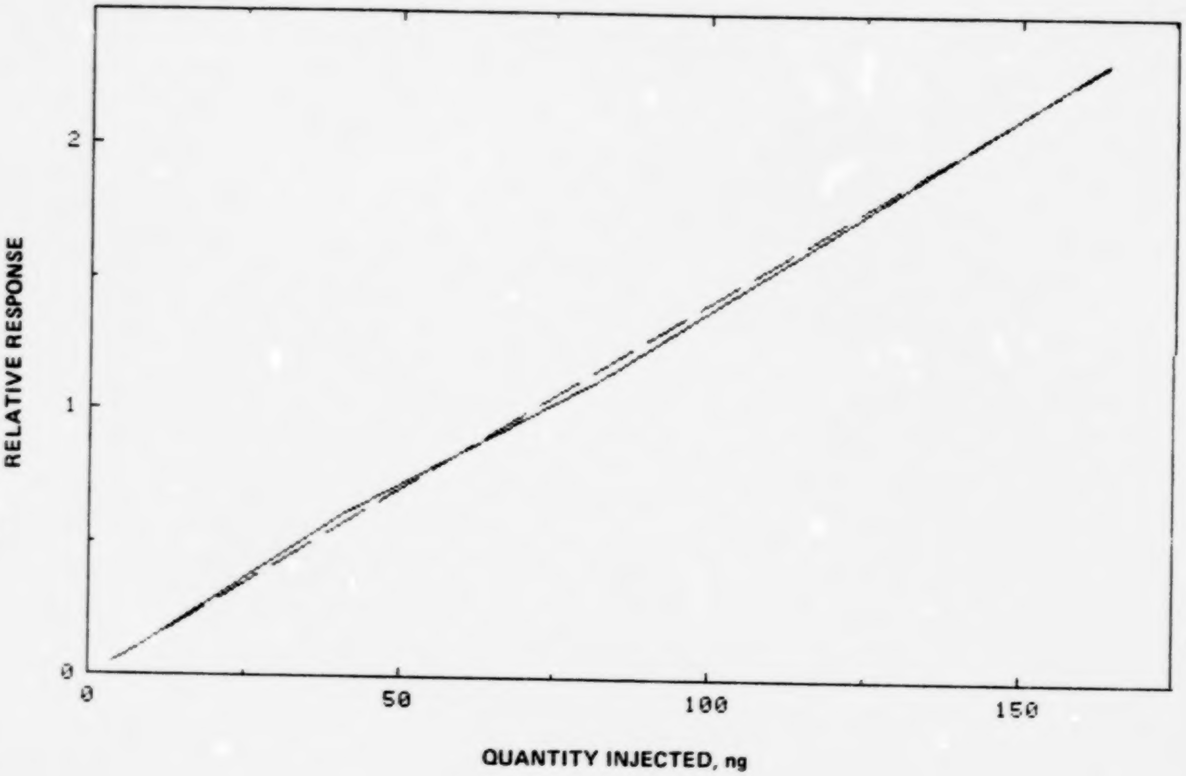
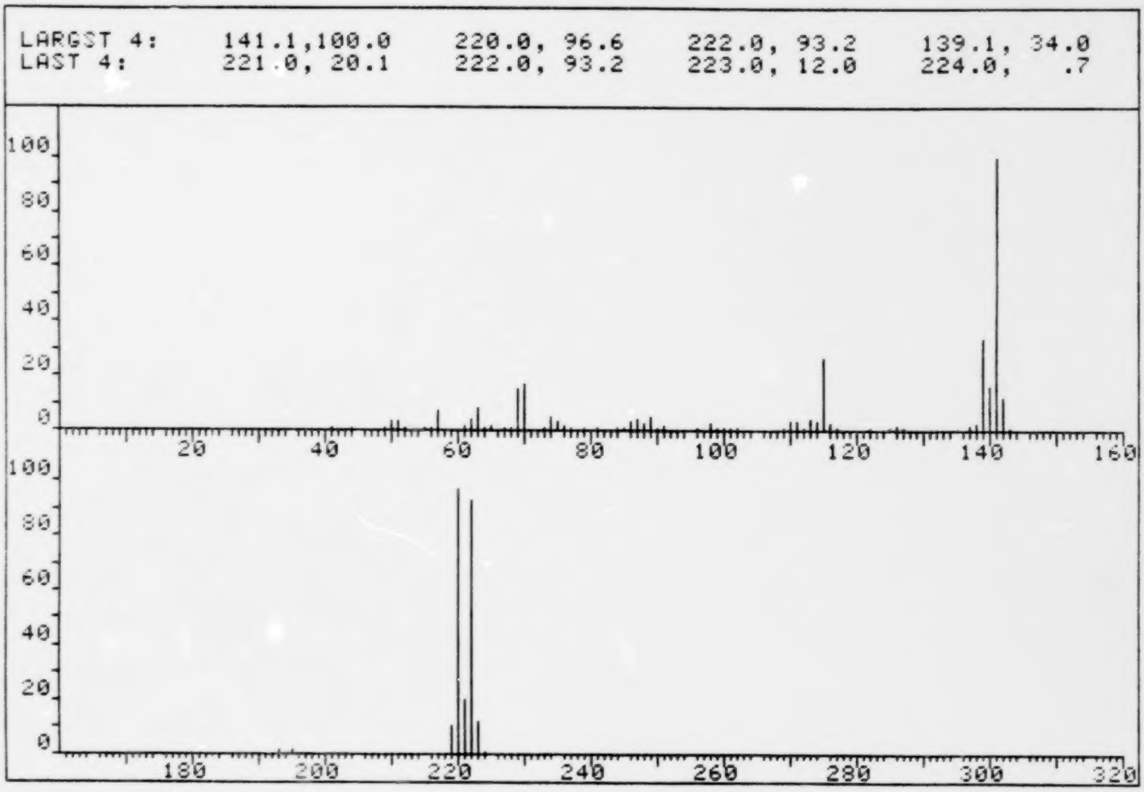


Figure B-39. Calibration curve for the determination of 3-bromoquinoline by GC/MS.

Range: 4.0 - 160 ng
Correlation coefficient: 0.999
Slope: 1.4 x 10^{-2}
y-Intercept: -5.6 x 10^{-3}



```
LARGST 4:     141.1,100.0     220.0, 96.6     222.0, 93.2     139.1, 34.0
LAST 4:       221.0, 20.1     222.0, 93.2     223.0, 12.0     224.0,    .7
```

Figure B-40. Mass spectrum for 1-bromo-4-methylnaphthalene.

TABLE B-18. KEY IONS AND RELATIVE ABUNDANCES FOR 1-BROMO-4-METHYLNAPHTHALENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	.2	70	16.7	98	2.3	127	.4
44	.1	73	.5	99	1.2	137	1.7
49	.2	74	5.0	100	.6	138	2.5
50	3.2	75	3.8	101	.4	139	34.0
51	3.2	76	1.8	102	.9	140	15.9
52	.7	77	.6	109	.6	141	100.0
55	1.1	79	.3	110	3.5	142	11.9
56	1.0	81	.3	111	3.8	143	.7
57	6.9	84	.1	112	.5	193	1.0
61	1.4	85	1.1	113	4.1	195	.9
62	4.1	86	3.1	114	3.9	219	9.9
63	8.3	87	4.2	115	26.2	220	96.6
64	1.2	88	2.4	116	2.5	221	20.1
65	1.9	89	5.2	117	.2	222	93.2
67	.4	90	.5	122	.3	223	12.0
68	.2	91	1.4	125	.3	224	.7
69	15.2	96	1.0	126	1.9		

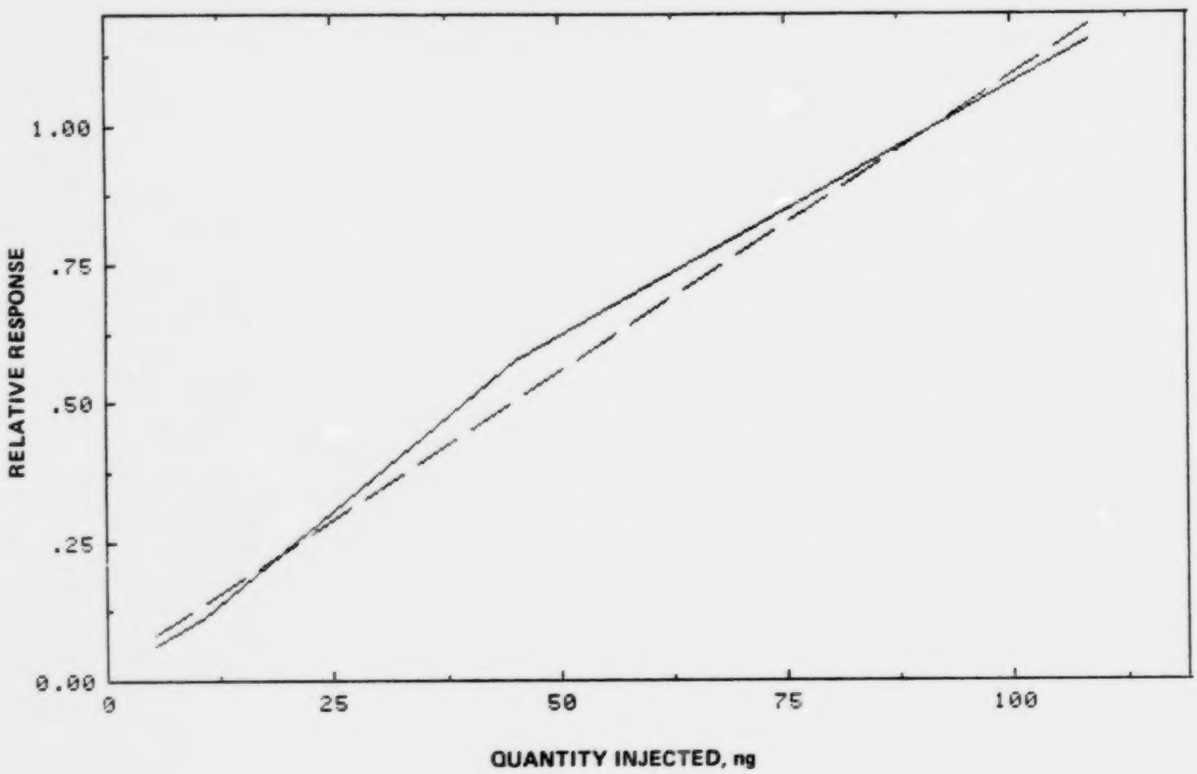
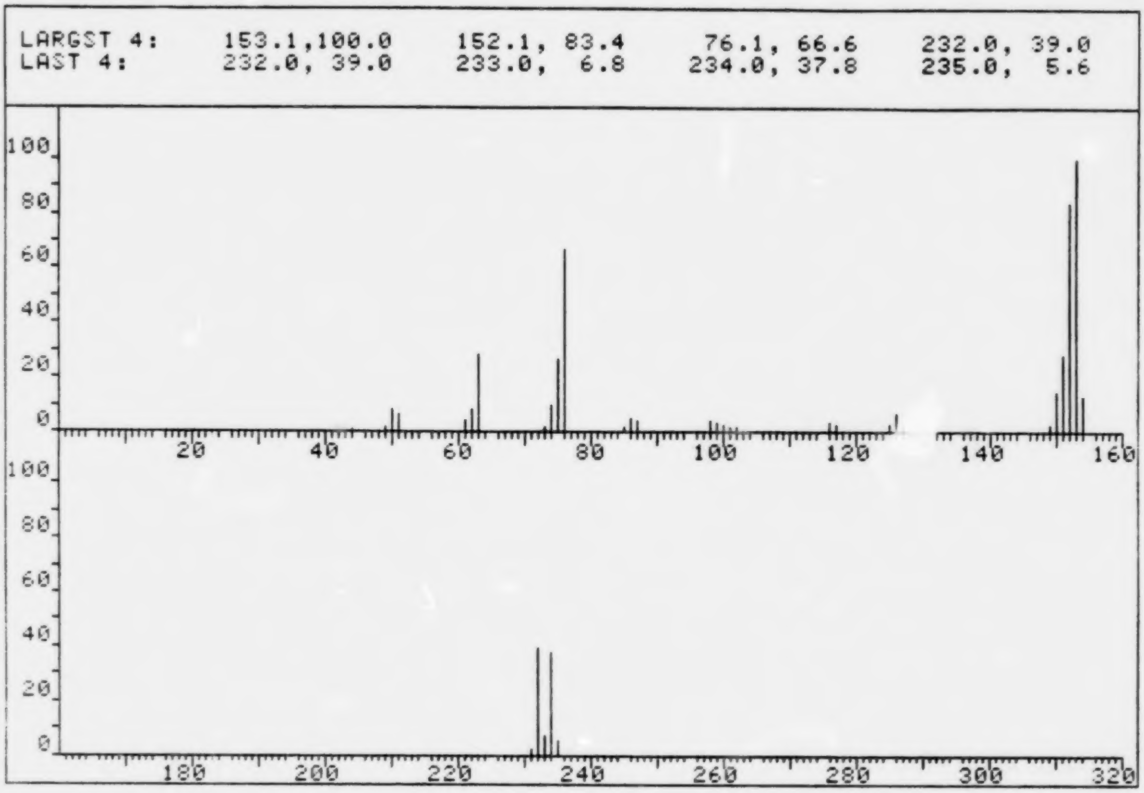


Figure B-41. Calibration curve for the determination of 1-bromo-4-methylnaphthalene by GC/MS.

Range: 5.0 - 110 ng
Correlation coefficient: 0.996
Slope: 1.1×10^{-2}
y-Intercept: 2.5×10^{-2}



```
LARGST 4:    153.1,100.0    152.1, 83.4     76.1, 66.6    232.0, 39.0
LAST 4:      232.0, 39.0    233.0,  6.8    234.0, 37.8    235.0,  5.6
```

Figure B-42. Mass spectrum for 5-bromoacenaphthene.

TABLE B-19. KEY IONS AND RELATIVE ABUNDANCES
FOR 5-BROMOACENAPHTHENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	.8	75	26.4	102	1.9	152	83.4
49	1.9	76	66.6	116	3.3	153	100.0
50	7.7	85	1.9	117	2.5	154	12.9
51	6.8	86	4.6	125	2.5	231	1.9
61	4.2	87	4.1	126	6.2	232	39.0
62	7.9	98	4.2	127	2.7	233	6.8
63	28.4	99	3.5	149	2.3	234	37.8
73	1.9	100	2.1	150	14.5	235	5.6
74	9.7	101	1.9	151	28.0		

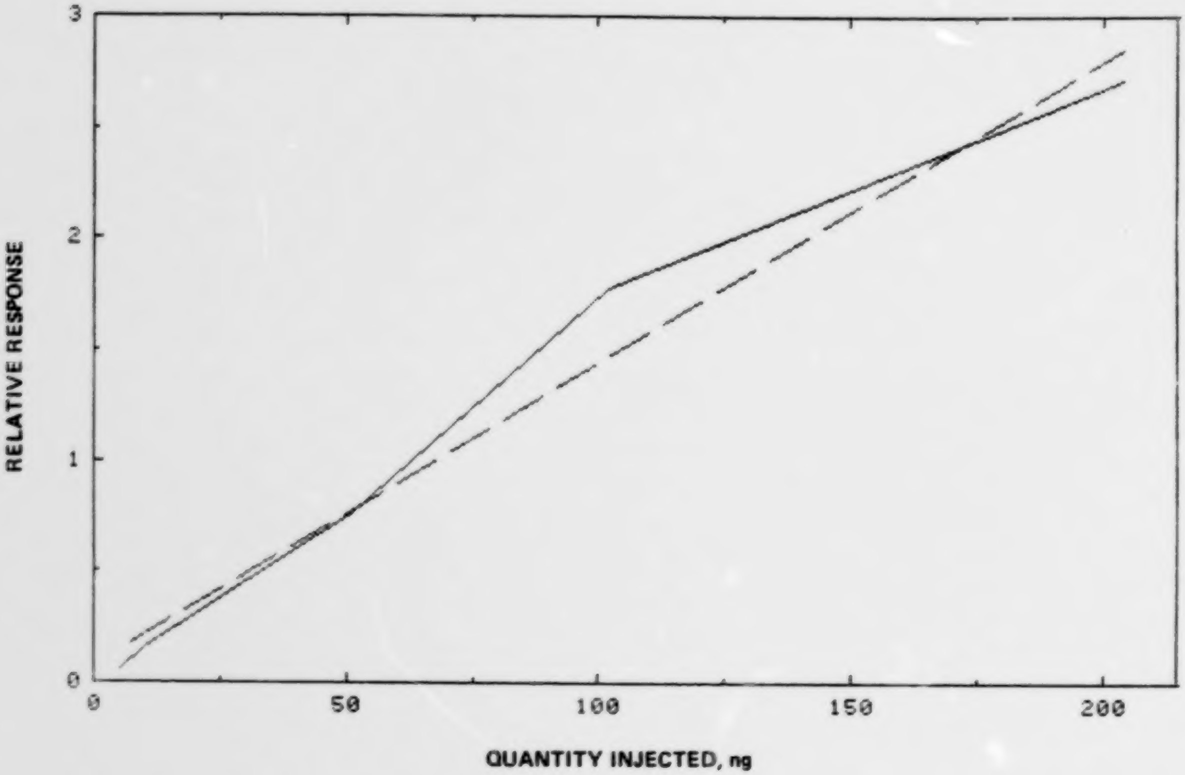


Figure B-43. Calibration curve for the determination of 5-bromoacenaphthene by GC/MS.

Range: 10 - 200 ng
Correlation coefficient: 0.988
Slope: 1.4×10^{-2}
y-Intercept: 7.9×10^{-2}

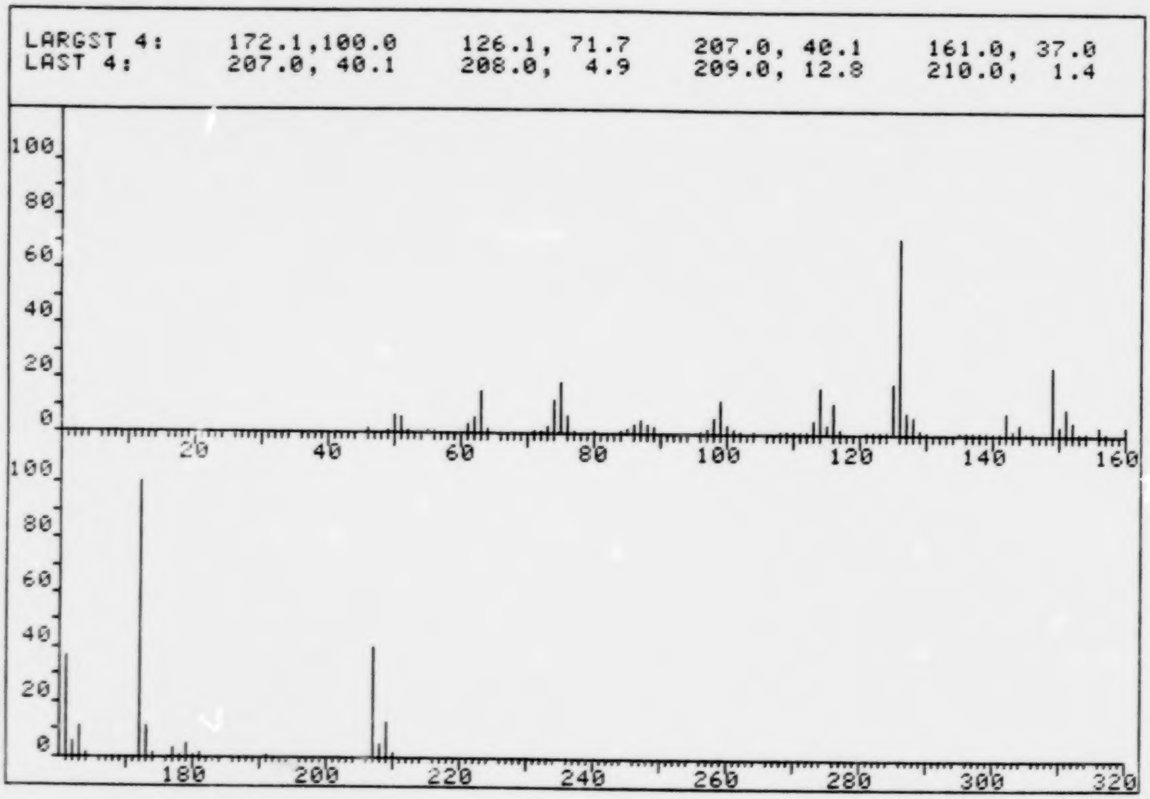


Figure B-44. Mass spectrum for 1-chloro-8-nitronaphthalene.

TABLE B-20. KEY IONS AND RELATIVE ABUNDANCES FOR 1-CHLORO-8-NITRONAPHTHALENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
46	1.5	88	3.0	124	1.1	153	.6
49	1.0	89	2.2	125	19.0	154	1.3
50	6.4	96	.4	126	71.7	156	3.2
51	5.5	97	1.3	127	8.6	157	.4
52	1.0	98	5.7	128	6.4	160	3.4
55	.4	99	12.0	129	1.3	161	37.0
56	.5	100	3.1	130	.6	162	5.5
61	2.8	101	1.4	135	.3	163	10.8
62	6.0	102	.5	136	.6	164	1.4
63	15.5	104	.7	137	.4	172	100.0
64	1.3	108	.4	138	.4	173	10.7
71	.6	109	.7	139	.4	174	1.2
72	.4	110	.8	140	.8	177	3.3
73	2.2	111	1.0	141	.9	178	.7
74	12.4	112	.5	142	8.1	179	4.4
75	19.0	113	4.8	143	1.5	180	.6
76	6.8	114	17.0	144	4.5	181	1.3
77	1.0	115	2.8	145	.8	191	.4
80	1.0	116	11.0	146	.4	207	40.1
84	.8	117	1.6	149	25.2	208	4.9
85	1.8	121	.6	150	3.0	209	12.8
86	3.7	122	1.2	151	9.8	210	1.4
87	4.7	123	1.2	152	4.7		

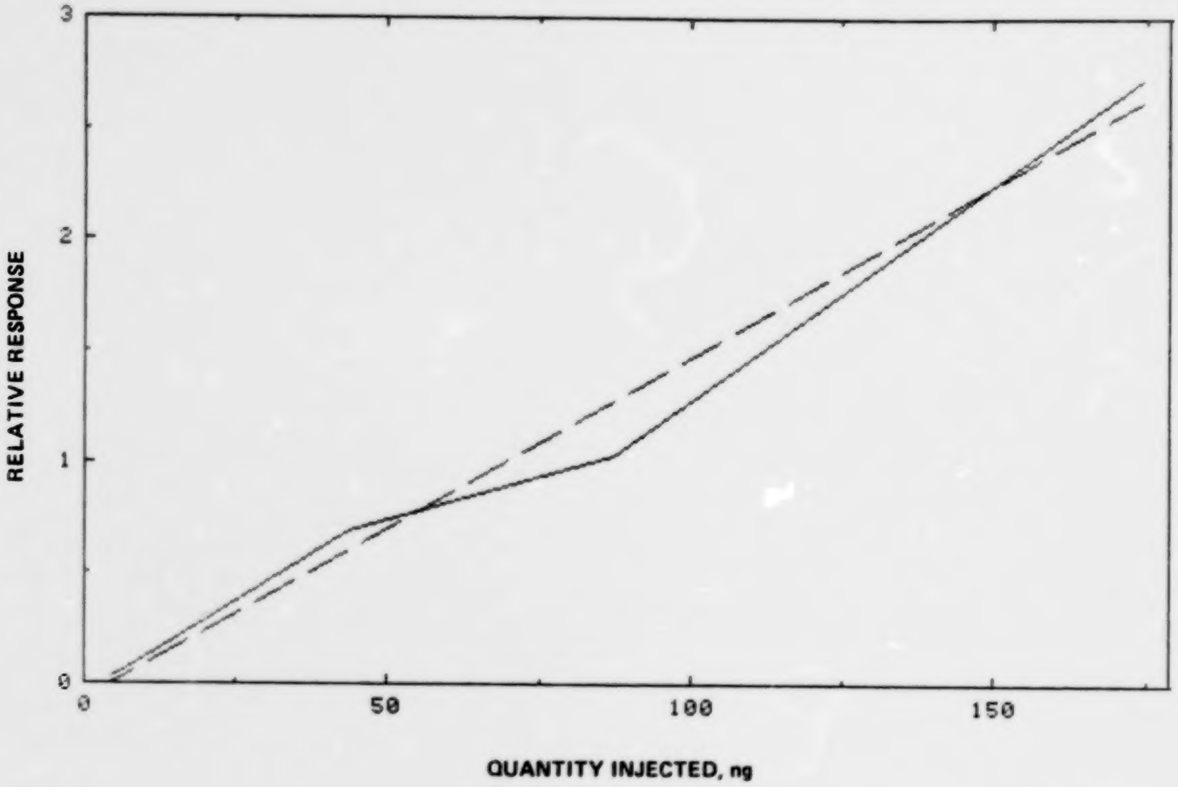
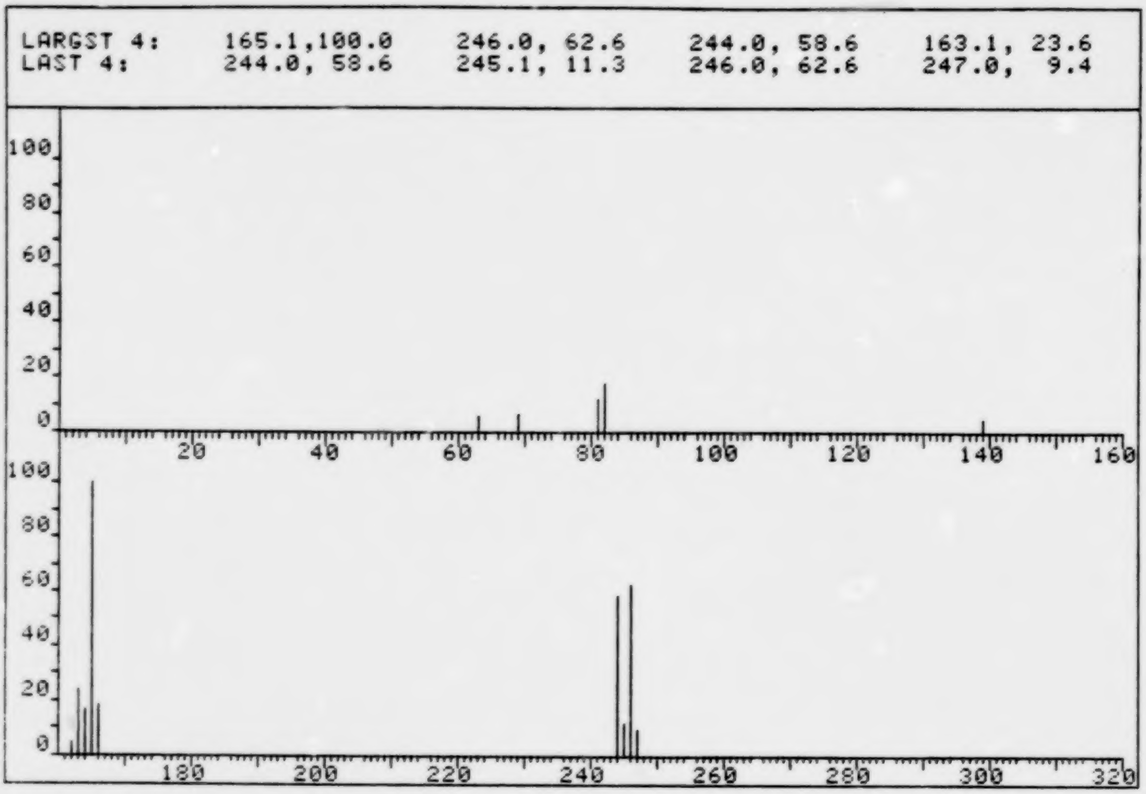


Figure B-45. Calibration curve for the determination of 1-chloro-8-nitronaphthalene by GC/MS.

Range: 9.0 - 170 ng
Correlation coefficient: 0.992
Slope: 1.5×10^{-2}
y-Intercept: -6.4×10^{-2}



```
LARGST 4:     165.1,100.0     246.0, 62.6     244.0, 58.6     163.1, 23.6
LAST 4:       244.0, 58.6     245.1, 11.3     246.0, 62.6     247.0,  9.4
```

Figure B-46. Mass spectrum for 2-bromofluorene.

TABLE B-21. KEY IONS AND RELATIVE ABUNDANCES FOR 2-BROMOFLUORENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
63	5.9	139	4.9	165	100.0	246	62.6
69	6.4	162	4.9	166	18.2	247	9.4
81	12.3	163	23.6	244	58.6		
82	18.2	164	16.3	245	11.3		

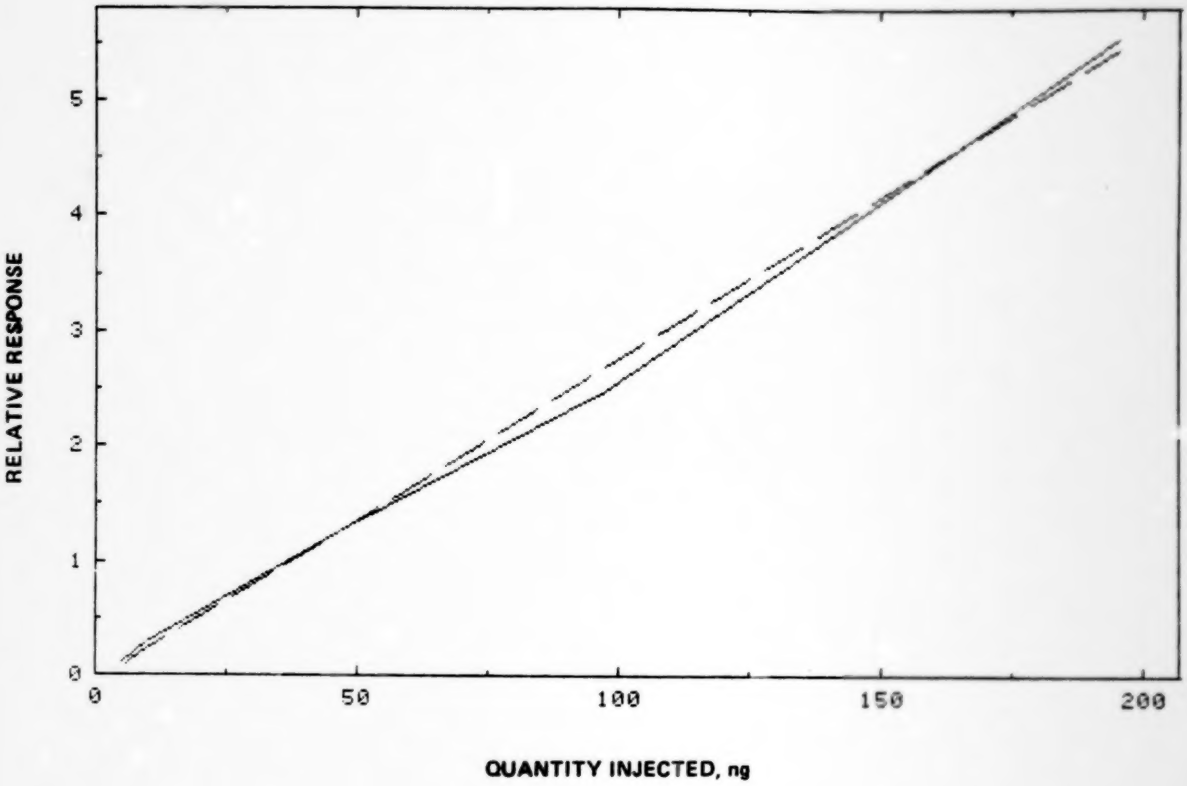
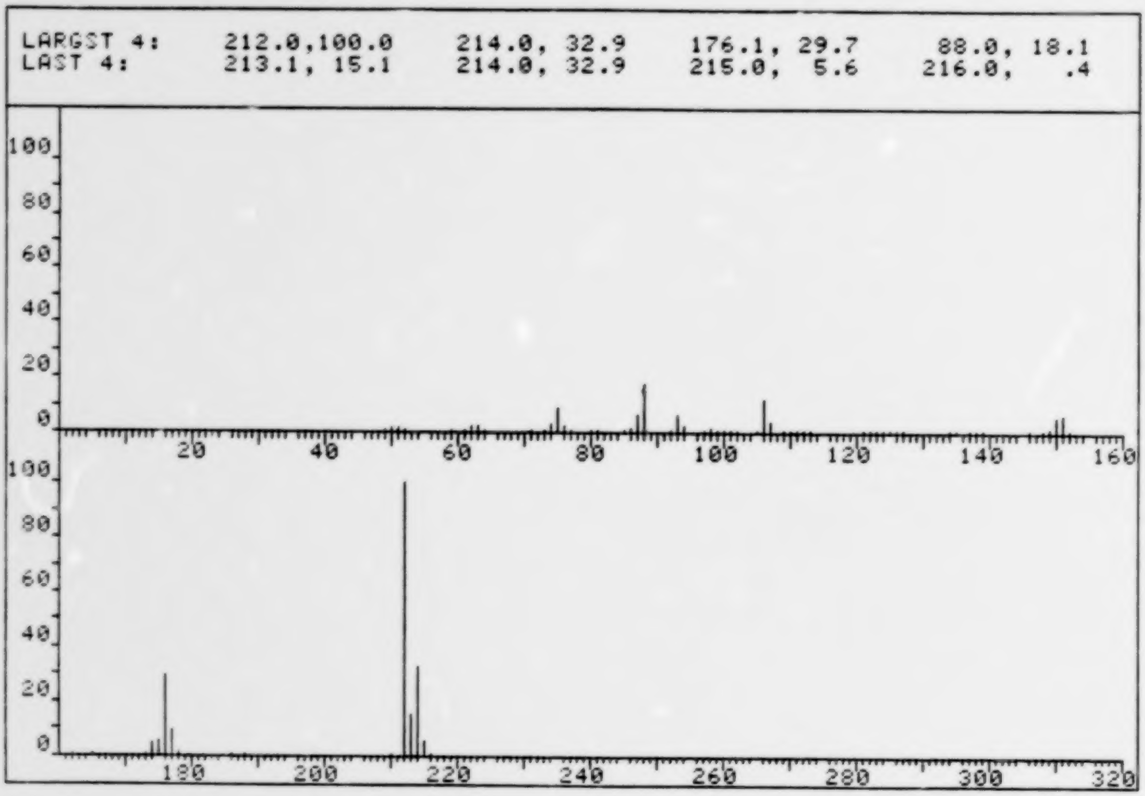


Figure B-47. Calibration curve for the determination of 2-bromofluorene by GC/MS.

Range: 5.0 - 200 ng
Correlation coefficient: 0.999
Slope: 2.8×10^{-2}
y-Intercept: -5.4×10^{-2}



```
LARGST 4:     212.0,100.0    214.0, 32.9    176.1, 29.7     88.0, 18.1
LAST 4:       213.1, 15.1    214.0, 32.9    215.0,  5.6    216.0,    .4
```

Figure B-48. Mass spectrum for 9-chloroanthracene.

TABLE B-22. KEY IONS AND RELATIVE ABUNDANCES FOR 9-CHLOROANTHRACENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
49	.2	86	1.4	113	.2	162	.2
50	1.4	87	6.8	121	.2	165	.2
51	1.4	88	18.1	122	.6	173	.5
52	.3	92	.3	123	.6	174	4.6
59	.2	93	6.8	124	.4	175	5.2
61	.7	94	2.6	125	.6	176	29.7
62	2.2	97	.3	126	1.3	177	9.4
63	2.1	98	1.8	127	.5	178	1.3
64	.3	99	1.2	134	.2	186	.5
71	.3	100	.4	135	.2	188	.2
71	.2	101	.3	137	.3	210	.4
73	.6	102	.3	138	.2	212	100.0
74	3.3	105	.6	139	.2	213	15.1
75	8.9	106	12.2	146	.2	214	32.9
76	2.4	107	4.1	148	.1	215	5.6
77	.7	109	.2	149	1.7	216	.4
80	.5	110	.6	150	5.6		
81	.3	111	.6	151	6.4		
85	.5	112	.1	152	1.0		

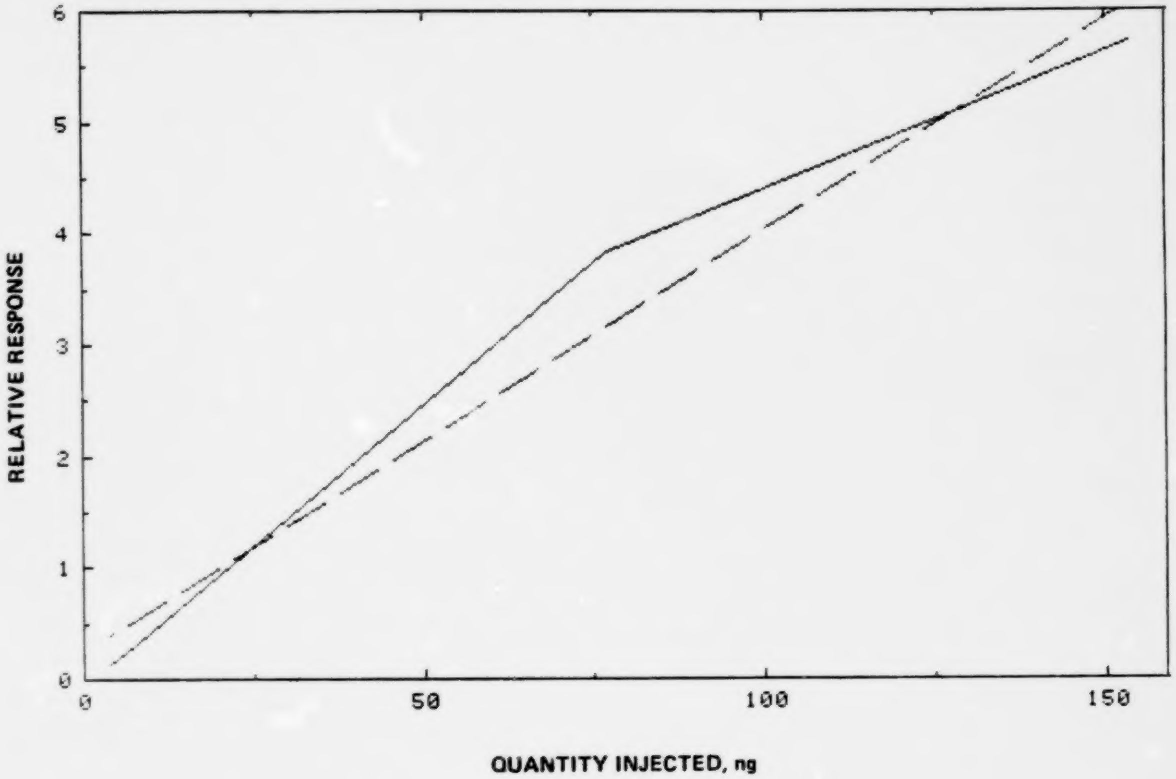


Figure B-49. Calibration curve for the determination of 9-chloroanthracene by GC/MS.

Range: 8.0 - 150 ng
Correlation coefficient: 0.985
Slope: 3.8×10^{-2}
y-Intercept: 2.5×10^{-1}

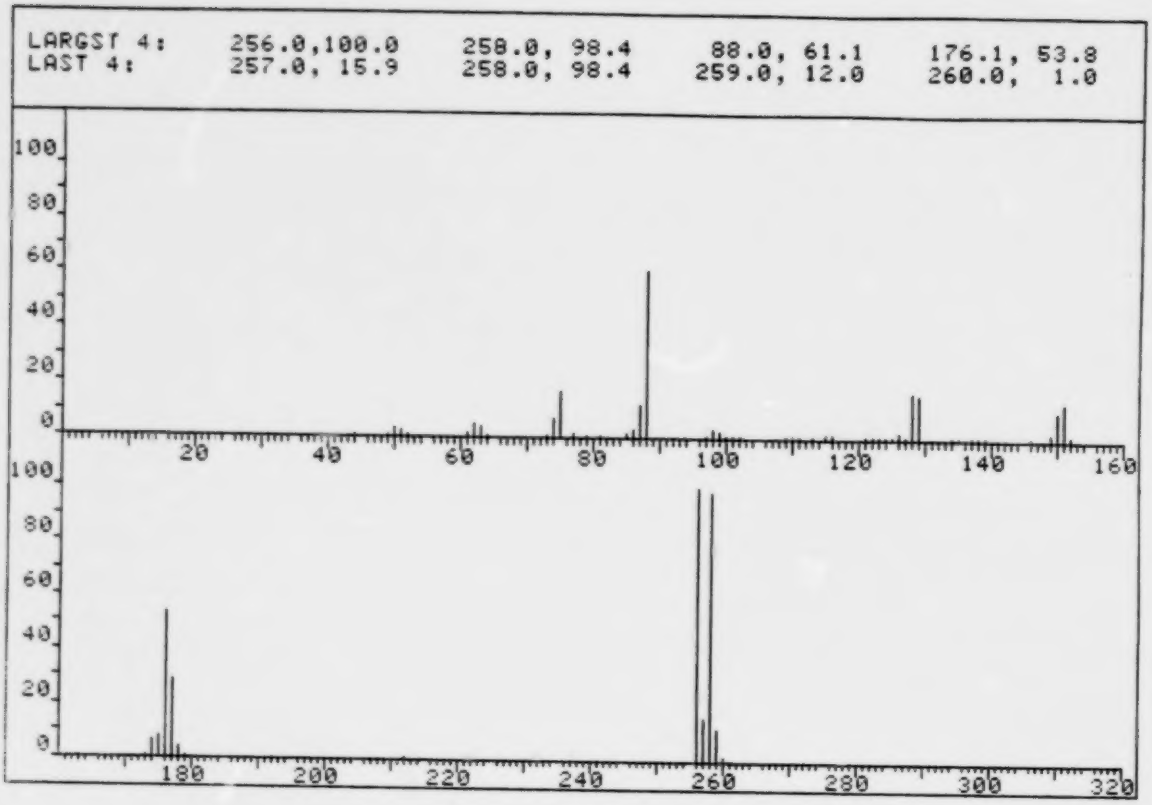


Figure B-50. Mass spectrum for 9-bromoanthracene.

TABLE B-23. KEY IONS AND RELATIVE ABUNDANCES FOR 9-BROMOANTHRACENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	.1	86	3.4	122	1.1	151	13.5
49	.3	87	11.9	123	.9	152	2.0
50	2.9	88	61.1	124	.5	173	.6
51	2.7	97	.4	125	1.1	174	6.4
52	.5	98	3.7	126	2.1	175	7.9
61	1.5	99	2.3	127	1.2	176	53.8
62	4.9	100	1.1	128	16.5	177	29.0
63	4.3	101	.7	129	15.9	178	4.1
64	.4	102	.5	134	.4	179	.3
73	.9	109	.5	135	.4	212	.1
74	7.0	110	1.1	137	.5	256	100.0
75	17.4	111	1.2	138	.3	257	15.9
77	1.8	113	.3	139	.3	258	98.4
79	.3	115	2.0	146	.3	259	12.0
81	.3	116	2.0	149	2.7	260	1.0
85	1.4	121	.3	150	10.8		

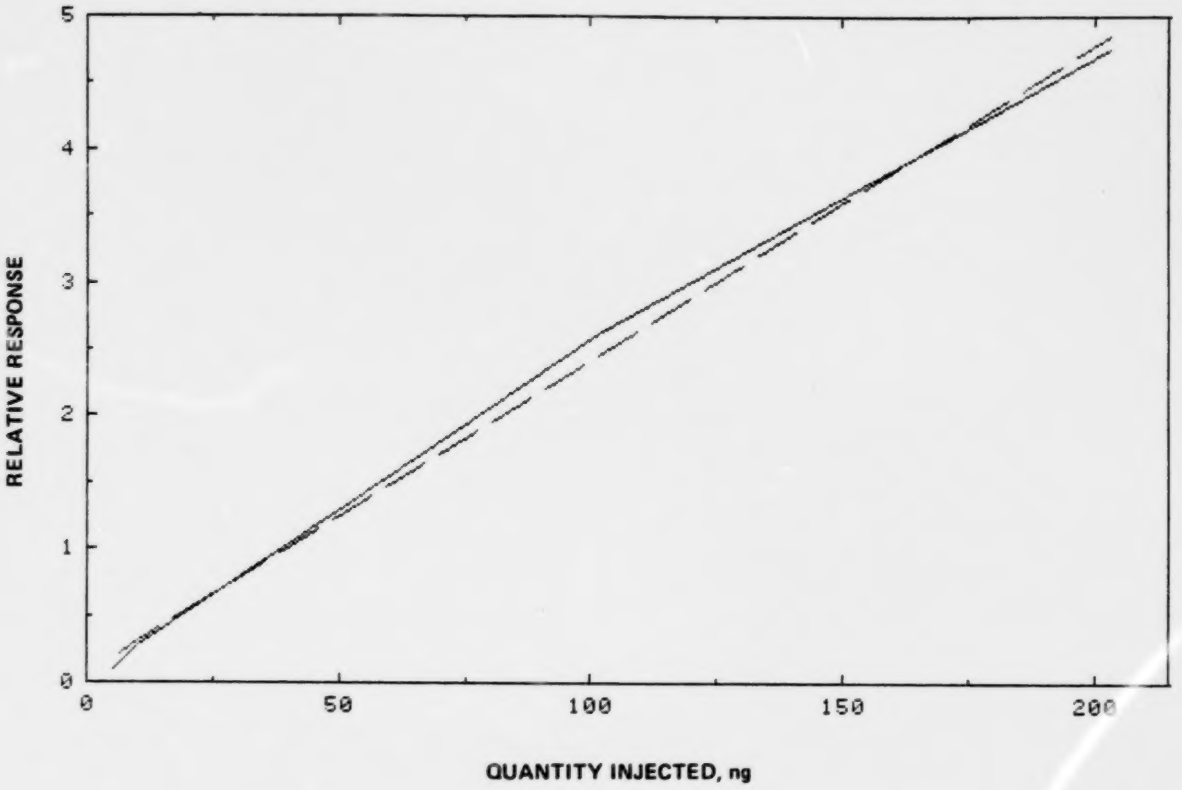
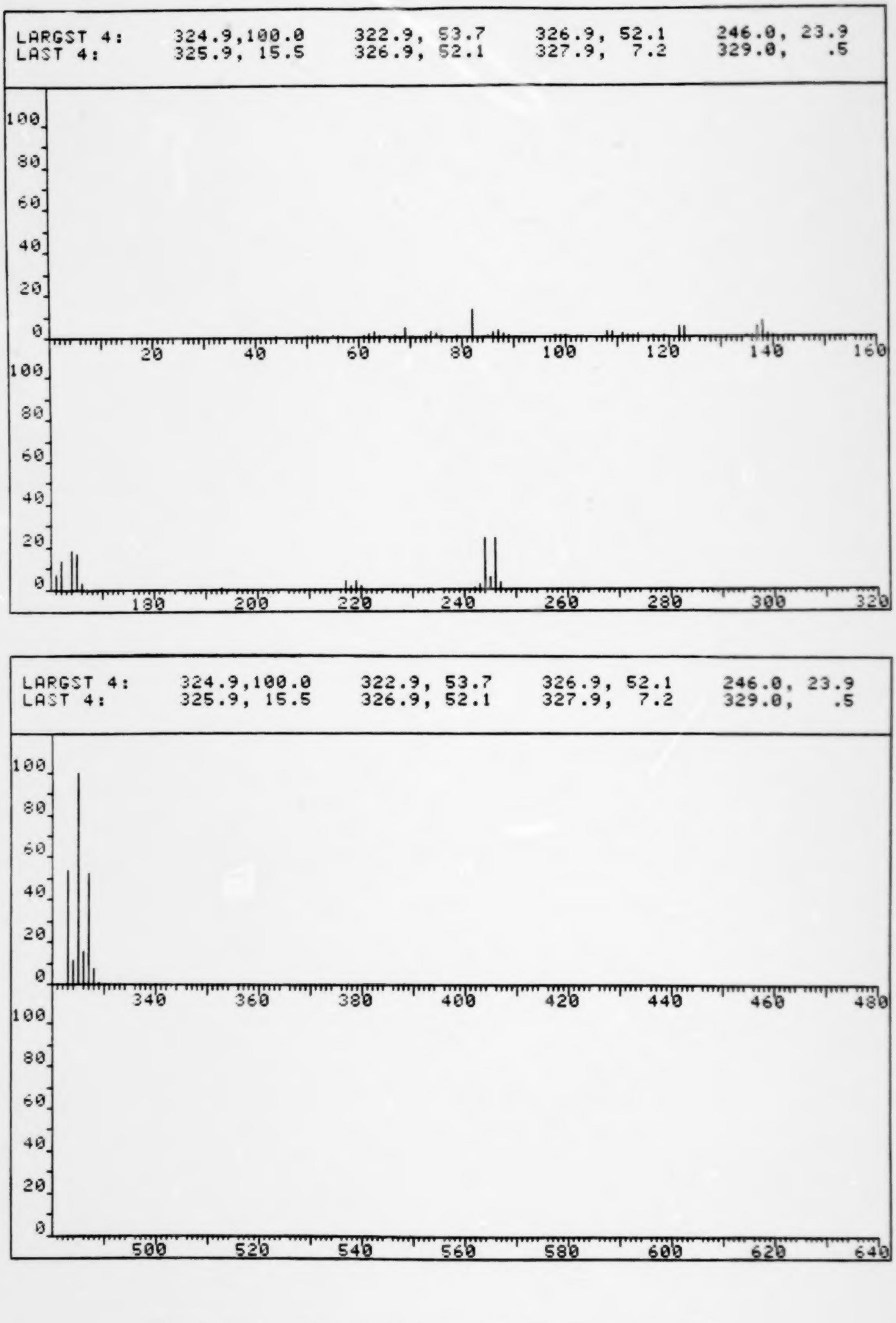


Figure B-51. Calibration curve for the determination of 9-bromoanthracene by GC/MS.

Range: 10 - 200 ng
Correlation coefficient: 0.998
Slope: 2.4×10^{-2}
y-Intercept: 7.0×10^{-2}



```
LARGST 4:     324.9,100.0     322.9, 53.7     326.9, 52.1     246.0, 23.9
LAST 4:       325.9, 15.5     326.9, 52.1     327.9,  7.2     329.0,    .5
```

```
LARGST 4:     324.9,100.0     322.9, 53.7     326.9, 52.1     246.0, 23.9
LAST 4:       325.9, 15.5     326.9, 52.1     327.9,  7.2     329.0,    .5
```

Figure B-52. Mass spectrum for 3,6-dibromocarbazole.

TABLE B-24. KEY IONS AND RELATIVE ABUNDANCES FOR 3,6-DIBROMOCARBAZOLE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	.1	79	.4	119	.6	219	3.6
50	1.1	82	12.8	122	4.7	220	1.0
51	.9	85	.8	123	4.8	242	.5
52	.7	86	2.3	135	.4	243	2.2
55	.9	87	2.8	136	.6	244	23.8
56	.8	88	1.9	137	5.3	245	5.2
61	1.0	89	.4	138	7.1	246	23.9
62	2.0	98	1.2	139	2.0	247	3.4
63	2.3	99	1.0	140	.6	323	53.7
64	.8	100	.8	161	6.8	324	11.4
65	.4	108	2.4	162	13.3	325	100.0
67	.6	109	2.3	164	18.3	326	15.5
69	4.2	111	1.3	165	16.7	327	52.1
73	.4	112	1.0	166	2.8	328	7.2
74	2.3	113	1.1	193	.4	329	.5
75	1.5	114	1.5	217	4.1		
76	.6	117	.5	218	1.0		

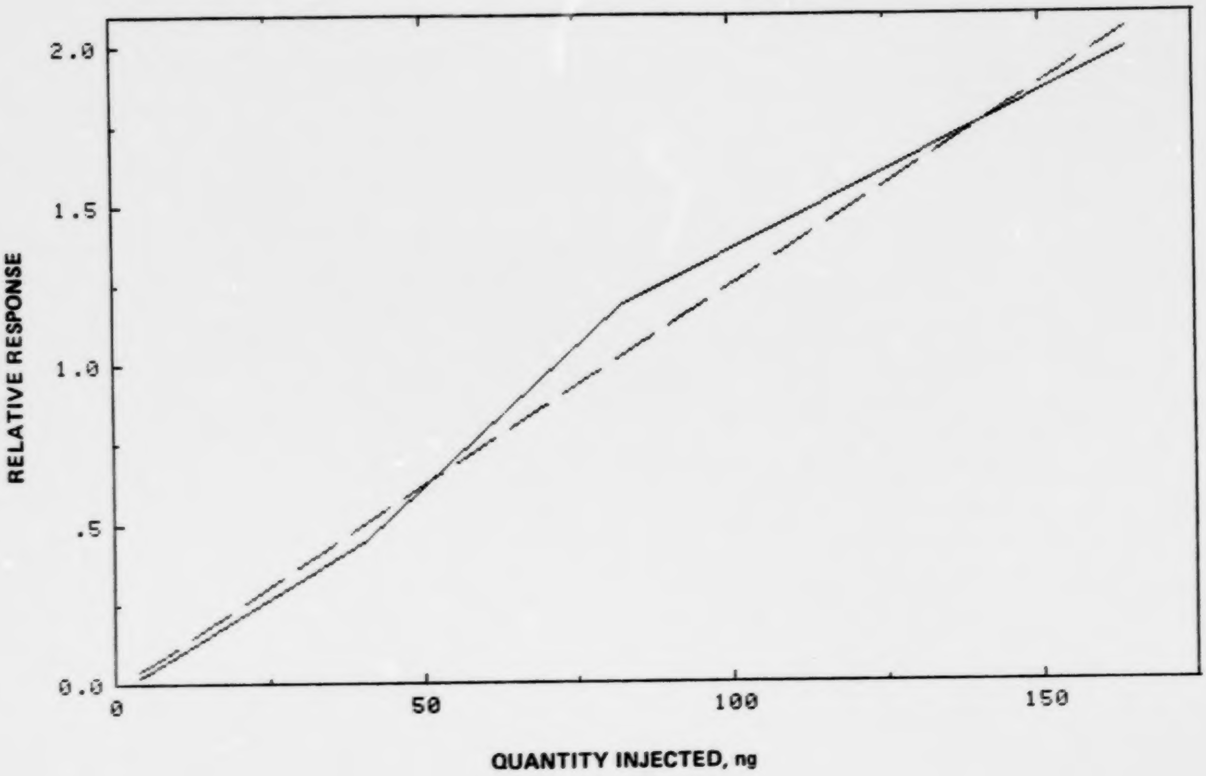


Figure B-53. Calibration curve for the determination of 3,6-dibromocarbazole by GC/MS.

Range: 4.0 - 160 ng
Correlation coefficient: 0.994
Slope: 1.3×10^{-2}
y-Intercept: -1.1×10^{-2}

TABLE B-25. KEY IONS AND RELATIVE ABUNDANCES FOR 1,2,3,4,6,8-HEPTACHLORODIBENZO-p-DIOXIN

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
47	1.1	120	3.2	183	3.0	331	2.1
49	1.3	121	2.1	184	1.4	333	2.6
51	1.2	130	3.4	188	1.1	335	1.9
60	1.8	131	4.6	190	1.4	337	1.3
61	2.6	132	3.0	191	.7	345	1.2
63	1.9	133	2.2	192	1.2	352	2.1
65	.7	134	1.3	193	1.0	354	3.8
71	2.0	141	1.5	194	1.2	356	1.6
72	1.1	142	8.3	195	1.4	359	13.1
73	1.9	143	4.4	211	5.9	361	27.8
77	2.1	144	5.2	212	12.9	362	3.5
78	1.3	145	3.0	213	11.0	363	22.9
83	2.3	146	1.3	214	7.3	364	3.2
84	4.6	147	3.1	215	2.7	365	10.1
85	1.5	148	5.1	216	1.6	366	1.5
86	1.7	149	10.5	226	4.1	367	2.9
87	3.4	150	5.3	228	3.0	387	2.9
88	1.1	151	1.7	230	1.3	389	4.8
89	.6	153	2.7	249	1.1	390	1.4
95	3.5	154	1.6	261	1.9	391	4.7
96	3.6	155	3.7	263	2.7	392	1.1
97	2.3	156	2.3	265	1.9	393	2.2
98	1.6	157	1.9	289	2.1	422	40.7
106	1.3	158	1.2	291	2.7	424	100.0
107	3.7	163	1.1	293	1.5	426	98.9
108	8.3	165	2.9	296	10.1	427	13.8
109	1.9	167	3.0	298	14.6	428	50.0
110	3.4	169	1.2	299	2.8	429	7.2
111	3.8	176	3.1	300	10.1	430	16.6
113	2.7	177	5.9	301	1.1	431	2.6
114	1.4	178	4.0	302	3.3	432	4.1
115	1.4	179	3.6	324	1.4		
118	4.2	180	5.0	326	1.9		
119	2.5	181	3.7	328	1.8		

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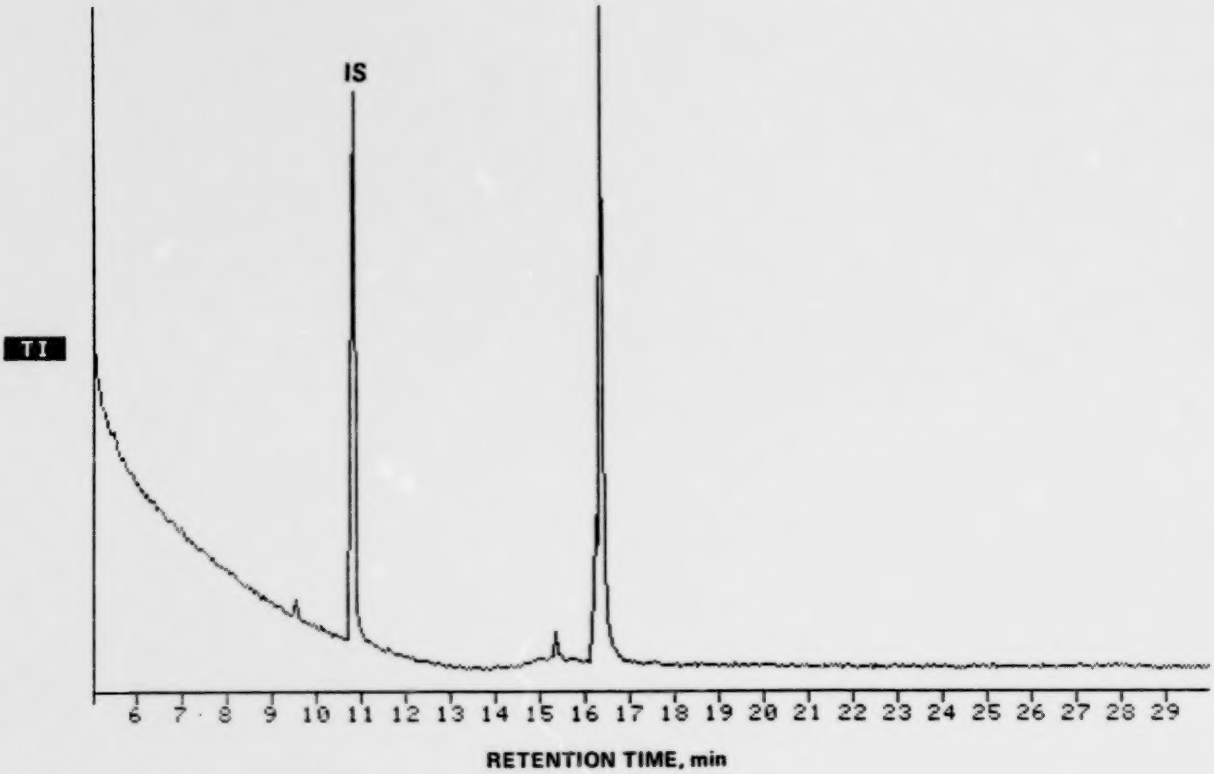
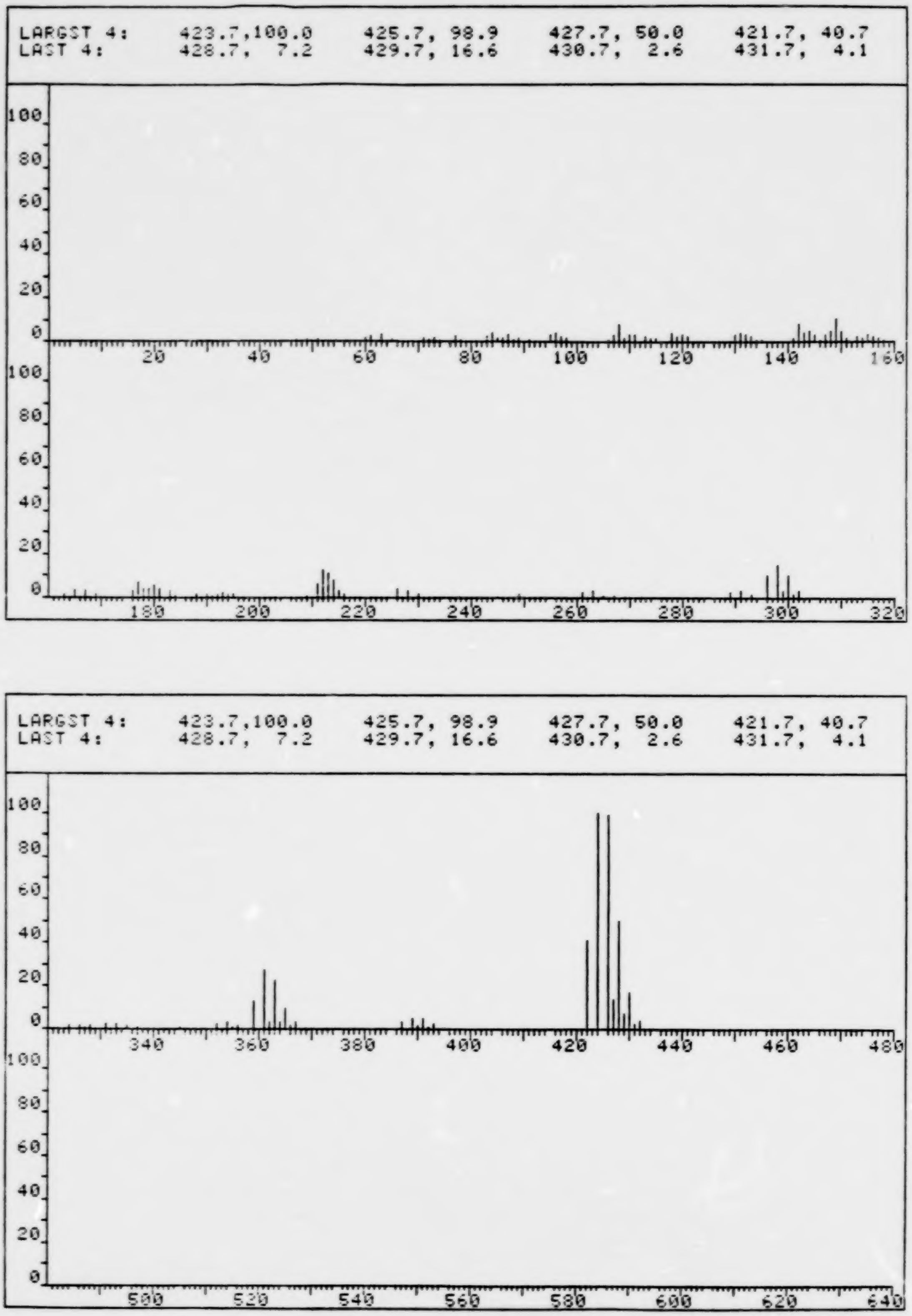


Figure B-54. Chromatogram by GC/MS for 1,2,3,4,6,7,8-heptachlorodibenzo-*p*-dioxin by GC/MS. (t_R = 16.4 min).

Temperature program:
60 °C, 3 min isothermal
60 to 300 °C at 20 °C/min
300 °C, 10 min isothermal



```
LARGST 4:     423.7,100.0     425.7, 98.9     427.7, 50.0     421.7, 40.7
LAST 4:       428.7,   7.2    429.7, 16.6     430.7,  2.6     431.7,  4.1
```

```
LARGST 4:     423.7,100.0     425.7, 98.9     427.7, 50.0     421.7, 40.7
LAST 4:       428.7,   7.2    429.7, 16.6     430.7,  2.6     431.7,  4.1
```

Figure B-55. Mass spectrum for 1,2,3,4,6,7,8-heptachloro-dibenzo-p-dioxin.

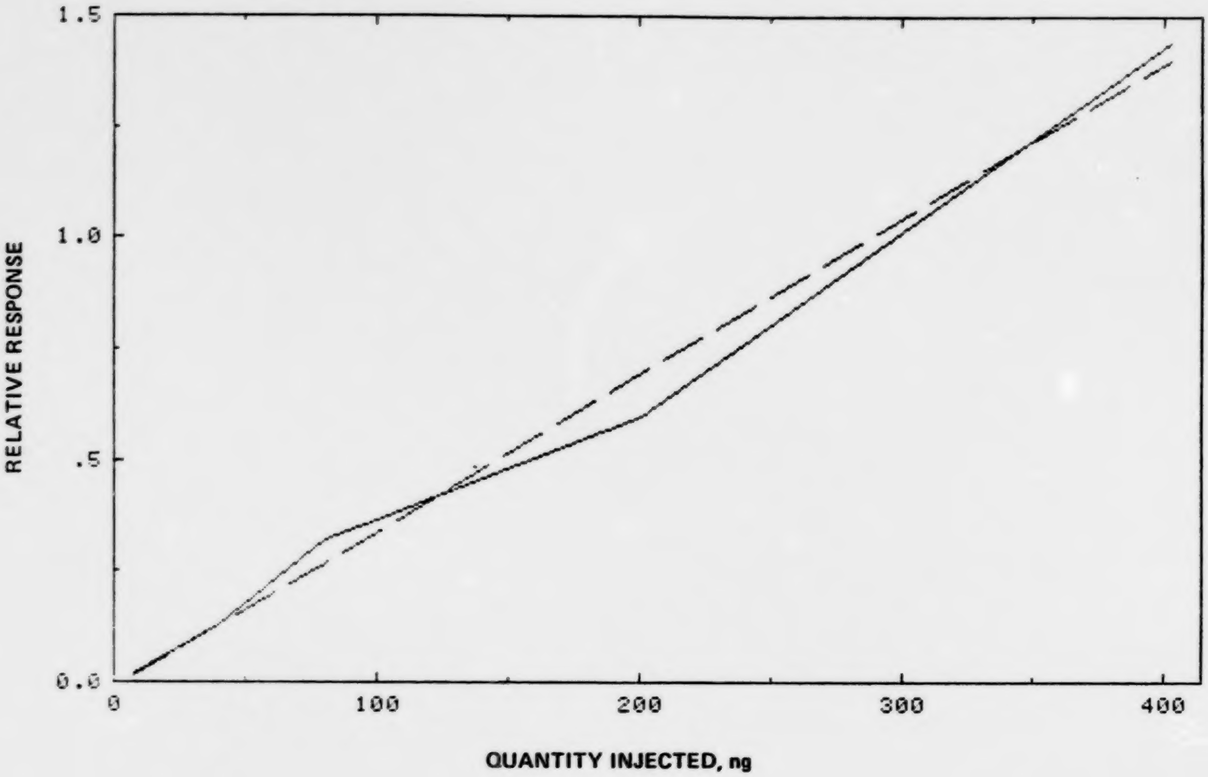


Figure B-56. Calibration curve for the determination of 1,2,3,4,6,7,8-heptachlorodibenzo-*p*-dioxin by GC/MS.

Range: 10 - 400 ng
Correlation coefficient: 0.995
Slope: 3.5×10^{-3}
y-Intercept: -1.4×10^{-2}

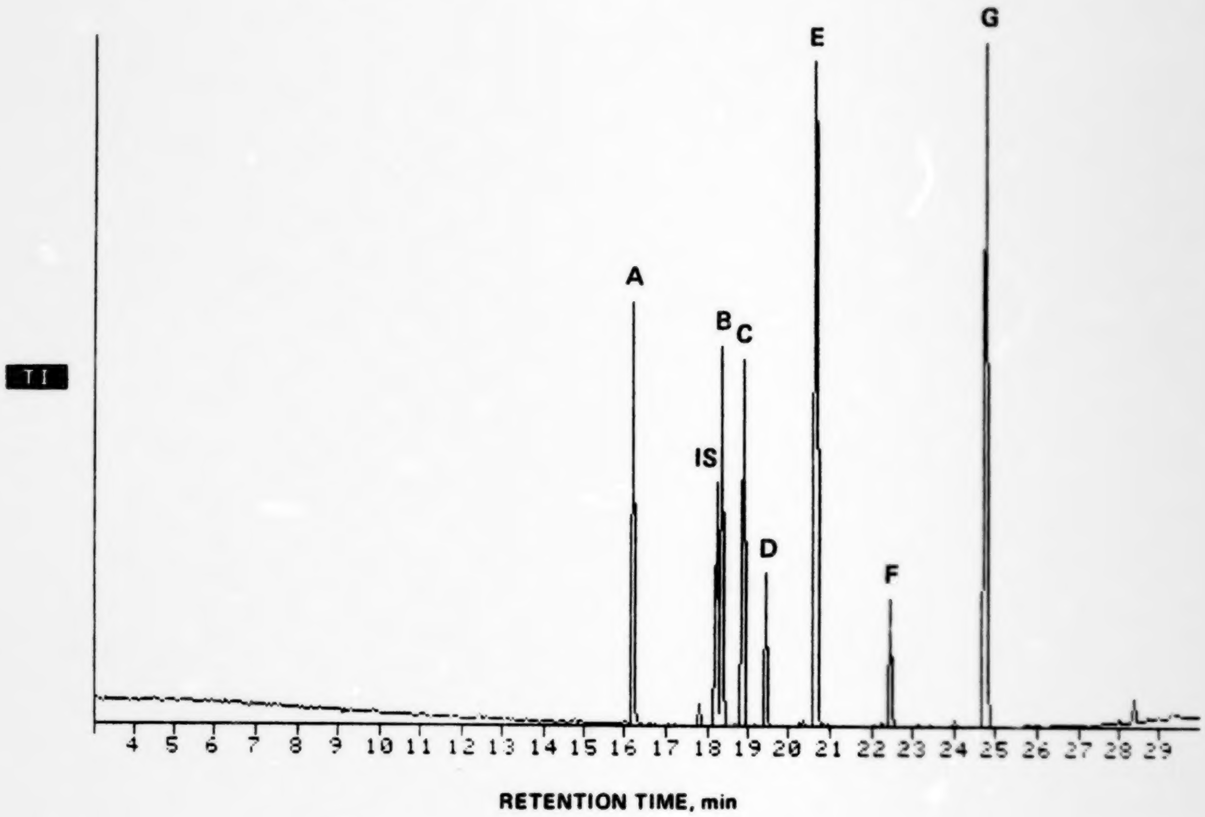
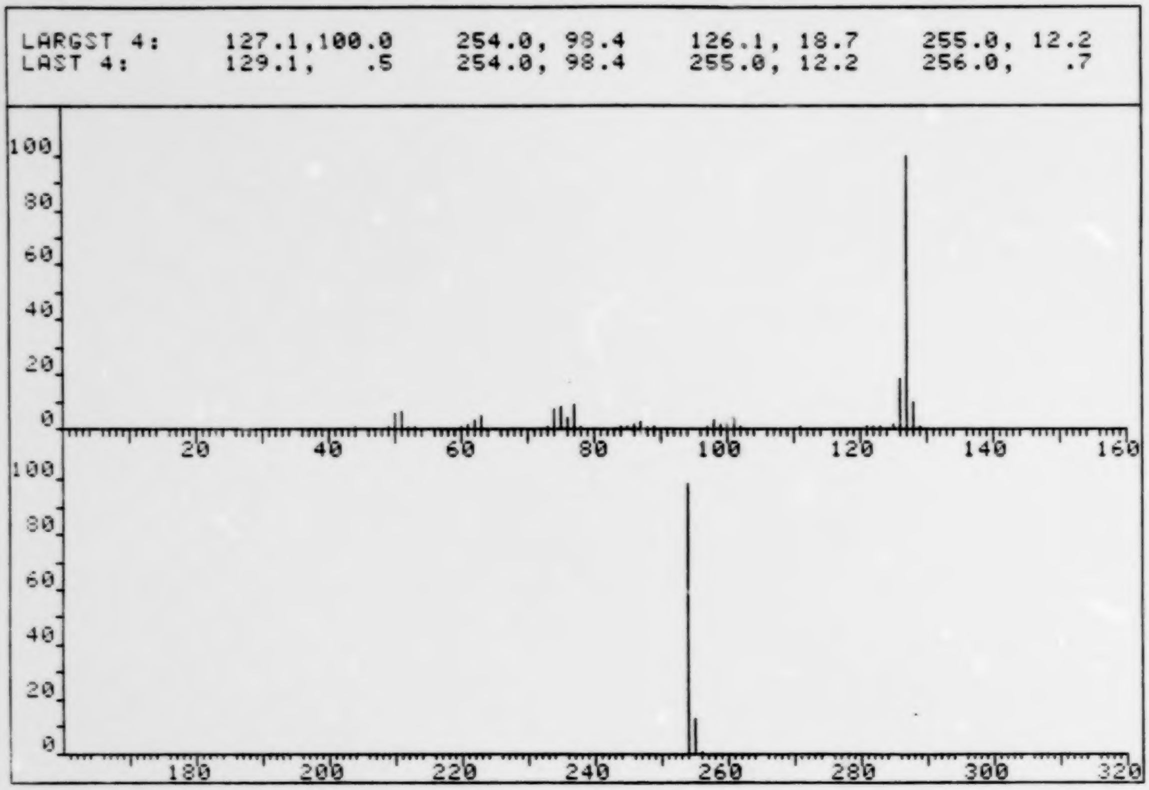


Figure B-57. Chromatogram by GC/MS for

(A) 1-iodonaphthalene(t_R = 16.1 min),
(B) 1-amino-4-chloronaphthalene(t_R = 18.4 min),
(C) 9-bromofluorene(t_R = 18.9 min),
(D) 1-amino-4-bromonaphthalene(t_R = 19.4 min),
(E) 2-chloroanthracene(t_R = 20.7 min),
(F) 9-(chloromethyl)anthracene(t_R = 22.4 min), and
(G) 9,10-dibromoanthracene(t_R = 24.8 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 1 min isothermal



```
LARGST 4:     127.1,100.0     254.0, 98.4     126.1, 18.7     255.0, 12.2
LAST 4:       129.1,    .5    254.0, 98.4     255.0, 12.2     256.0,    .7
```

Figure B-58. Mass spectrum for 1-iodonaphthalene.

TABLE B-26. KEY IONS AND RELATIVE ABUNDANCES FOR 1-IODONAPHTHALENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	.0	73	1.0	88	.3	122	.6
49	.7	74	7.5	89	.2	123	.2
50	5.7	75	7.6	97	.5	125	1.4
51	6.6	76	4.5	98	3.2	126	18.7
52	1.0	77	8.8	99	1.6	127	100.0
53	.3	78	.7	100	1.6	128	9.9
60	.2	84	.3	101	4.5	129	.5
61	1.6	85	.8	102	.4	254	98.4
62	2.9	86	1.8	111	.2	255	12.2
63	5.2	87	2.3	121	.3	256	.7

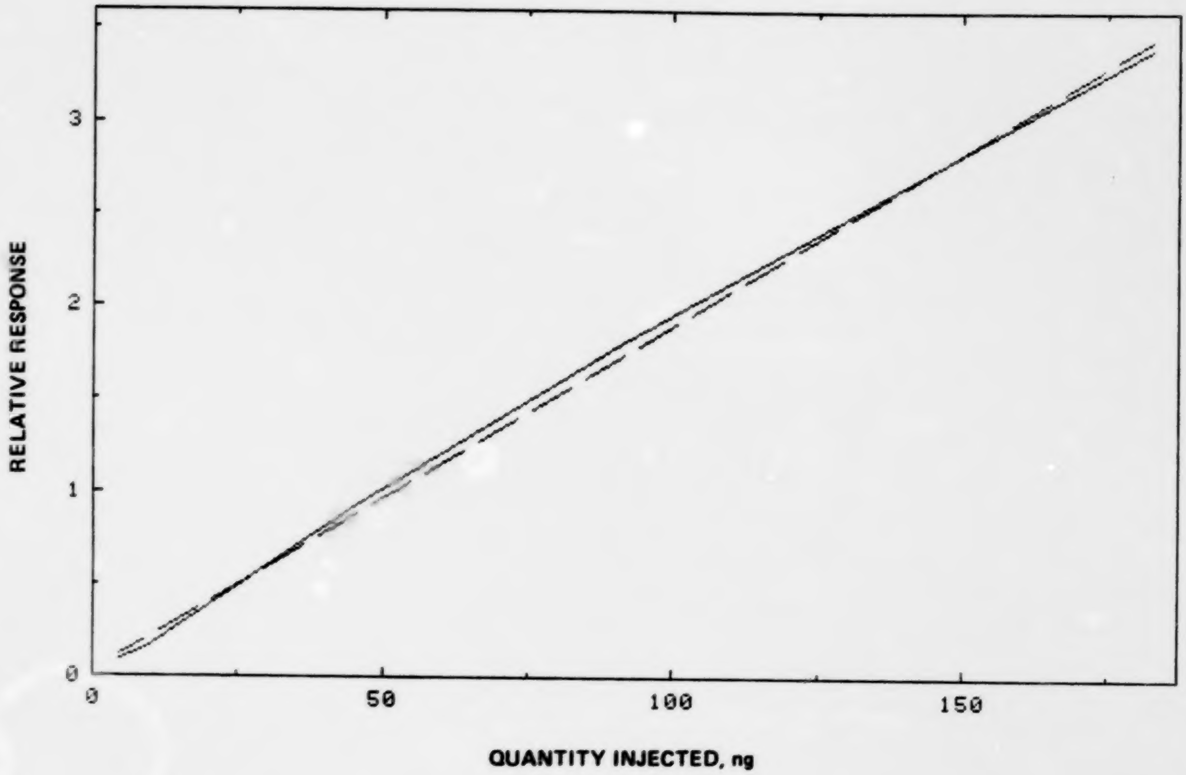
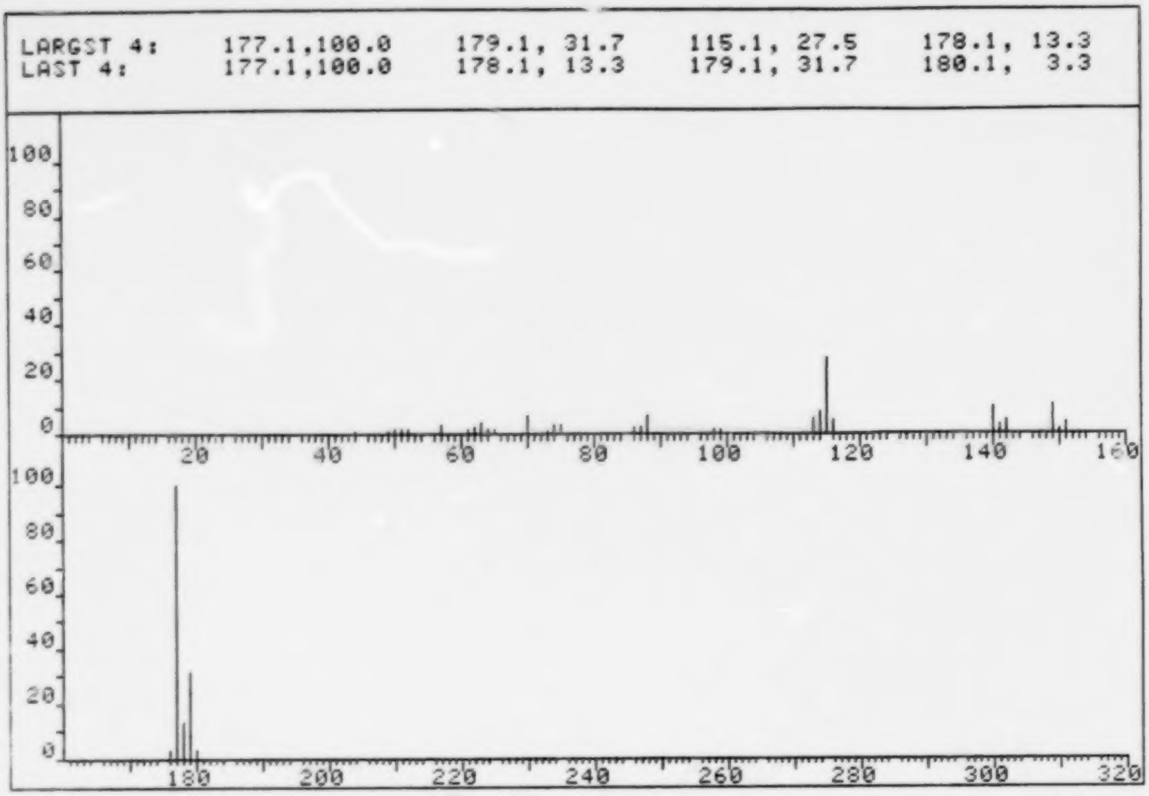


Figure B-59. Calibration curve for the determination of 1-iodonaphthalene by GC/MS.

Range: 5.0 - 180 ng
Correlation coefficient: 0.999
Slope: 1.9 x 10^{-2}
y-Intercept: 2.8 x 10^{-2}



```
LARGST 4:     177.1,100.0     179.1, 31.7     115.1, 27.5     178.1, 13.3
LAST 4:       177.1,100.0     178.1, 13.3     179.1, 31.7     180.1,  3.3
```

Figure B-60. Mass spectrum for 1-amino-4-chloronaphthalene.

TABLE B-27. KEY IONS AND RELATIVE ABUNDANCES FOR 1-AMINO-4-CHLORONAPHTHALENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	.3	64	1.5	98	1.4	149	10.6
49	1.3	65	1.4	99	1.4	150	1.9
50	1.8	70	6.3	113	5.9	151	4.3
51	1.9	73	1.3	114	7.8	176	3.1
52	1.4	74	2.8	115	27.5	177	100.0
57	2.8	75	2.9	116	4.9	178	13.3
61	1.4	86	2.6	140	10.1	179	31.7
62	2.6	87	2.4	141	2.9	180	3.3
63	4.6	88	6.3	142	5.2		

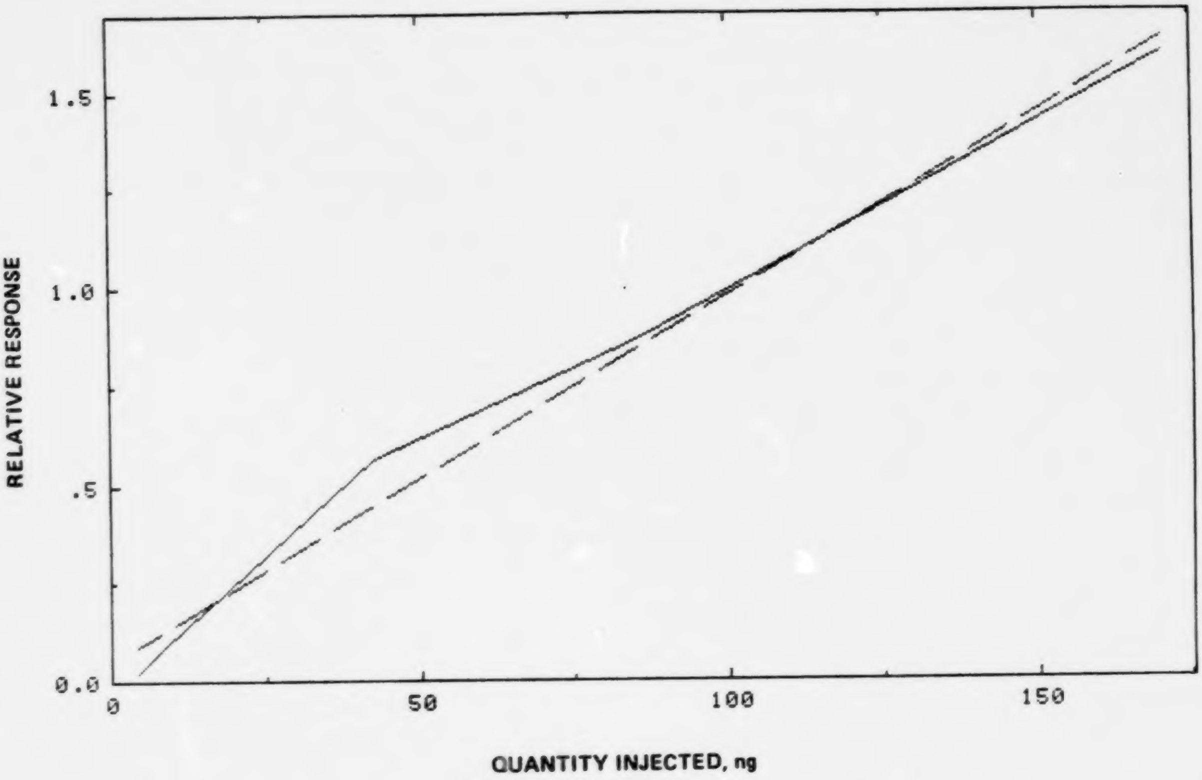
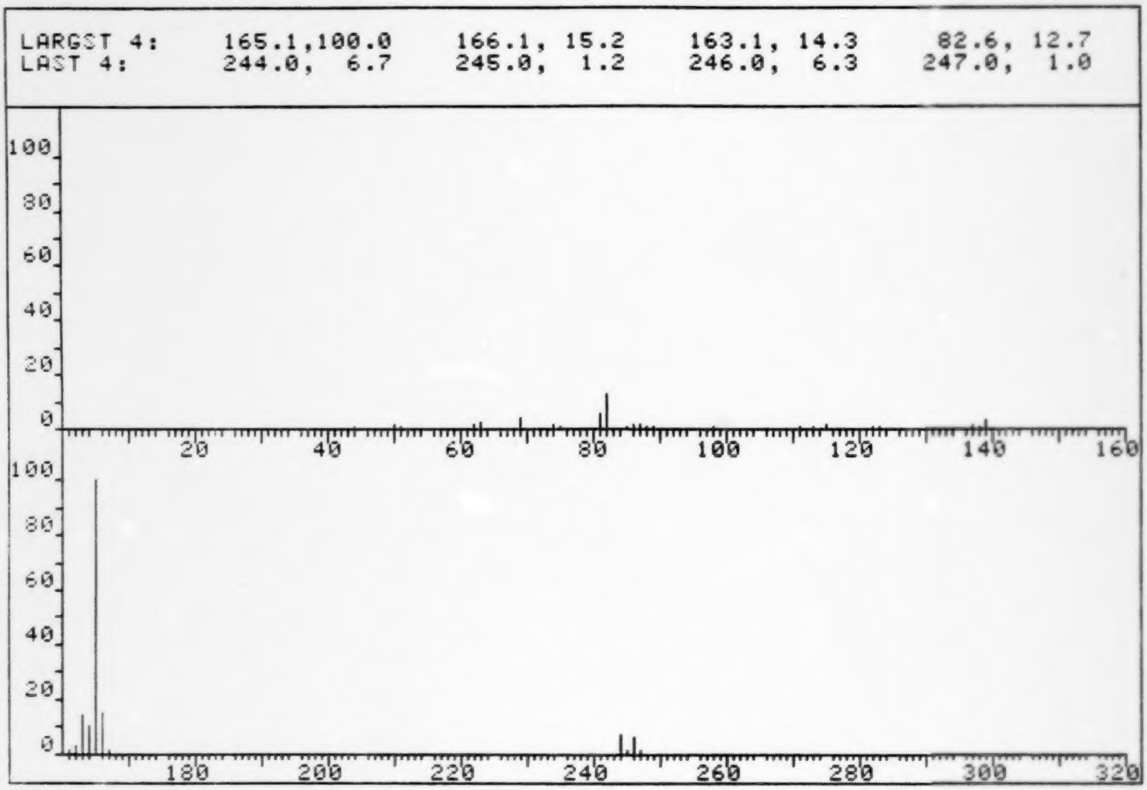


Figure B-61. Calibration curve for the determination of 1-amino-4-chloronaphthalene by GC/MS.

Range: 9.0 - 170 ng
Correlation coefficient: 0.993
Slope: 9.3×10^{-3}
y-Intercept: 5.1×10^{-2}



```
LARGST 4:     165.1,100.0     166.1, 15.2     163.1, 14.3      82.6, 12.7
LAST 4:       244.0,   6.7    245.0,  1.2     246.0,  6.3     247.0,  1.0
```

Figure B-62. Mass spectrum for 9-bromofluorene.

TABLE B-28. KEY IONS AND RELATIVE ABUNDANCES FOR 9-BROMOFLUORENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	.1	82	12.7	115	1.5	164	10.3
50	1.4	85	.7	122	1.0	165	100.0
51	1.0	86	1.8	123	.7	166	15.2
62	1.5	87	2.0	137	1.7	167	1.2
63	2.4	88	.9	138	1.0	244	6.7
69	4.6	89	1.0	139	3.3	245	1.2
74	1.8	98	1.2	161	1.2	246	6.3
75	1.2	111	.7	162	3.3	247	1.0
81	6.0	113	1.0	163	14.3		

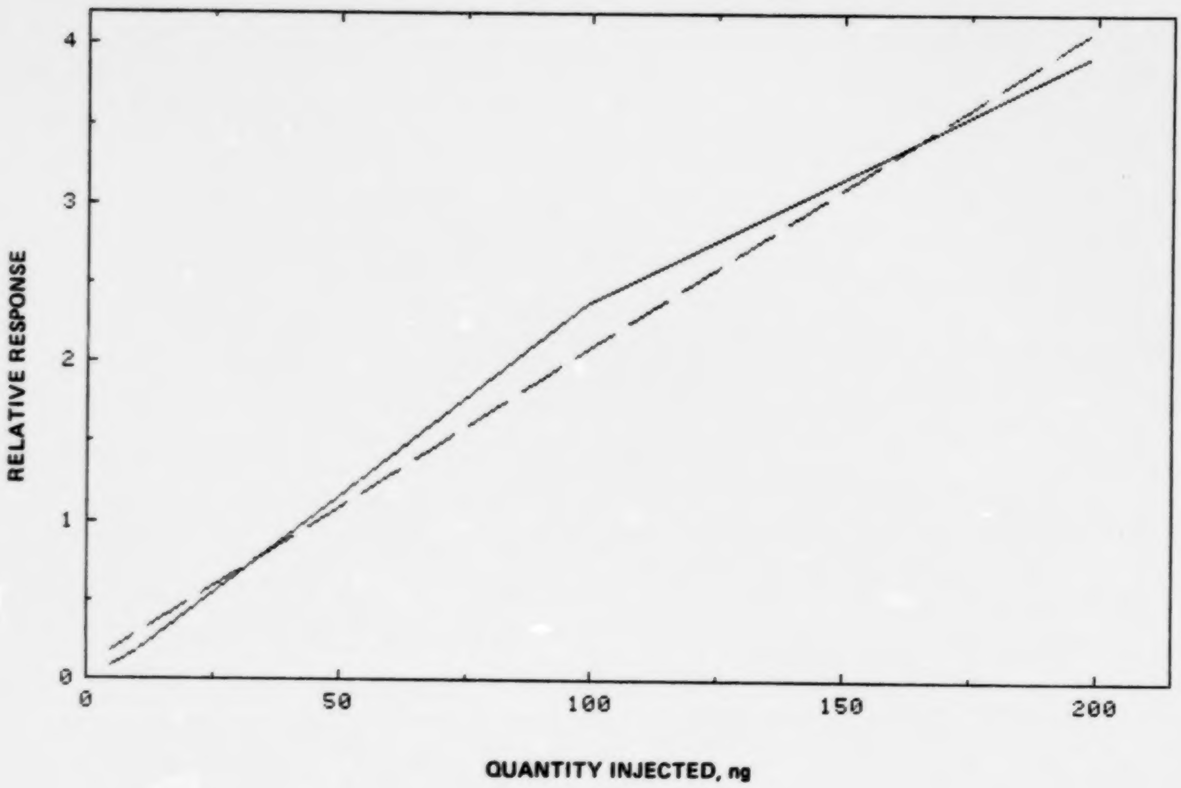
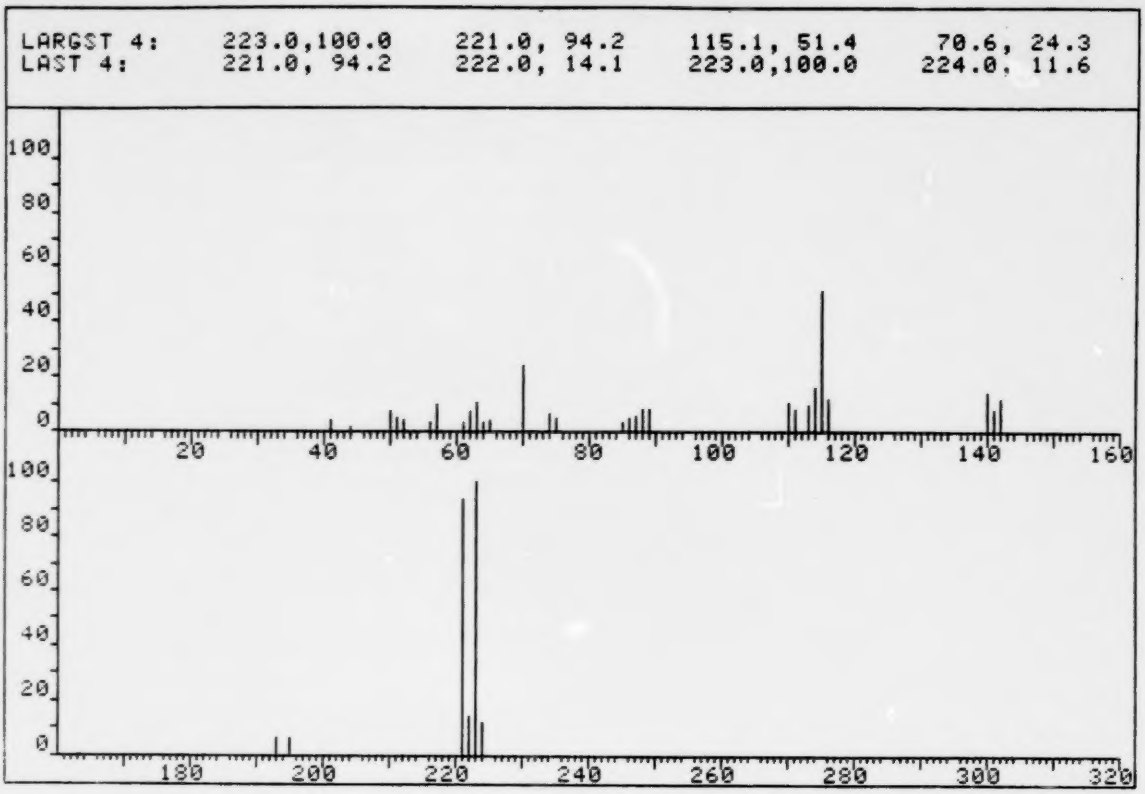


Figure B-63. Calibration curve for the determination of 9-bromofluorene by GC/MS.

Range: 10 - 200 ng
Correlation coefficient: 0.993
Slope: 2.0×10^{-2}
y-Intercept: 8.5×10^{-2}



```
LARGST 4:     223.0,100.0     221.0, 94.2     115.1, 51.4      70.6, 24.3
LAST 4:       221.0, 94.2     222.0, 14.1     223.0,100.0     224.0, 11.6
```

Figure B-64. Mass spectrum for 1-amino-4-bromonaphthalene.

TABLE B-29. KEY IONS AND RELATIVE ABUNDANCES FOR 1-AMINO-4-BROMONAPHTHALENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	4.0	63	10.5	88	8.0	141	8.0
44	1.8	64	3.6	89	8.3	142	12.3
50	6.9	65	4.3	110	10.5	193	5.8
51	5.1	70	24.3	111	8.0	195	6.2
52	4.3	74	6.2	113	10.1	221	94.2
56	3.6	75	5.1	114	15.9	222	14.1
57	9.8	85	3.3	115	51.4	223	100.0
61	3.6	86	5.1	116	12.0	224	11.6
62	7.2	87	5.8	140	14.9		

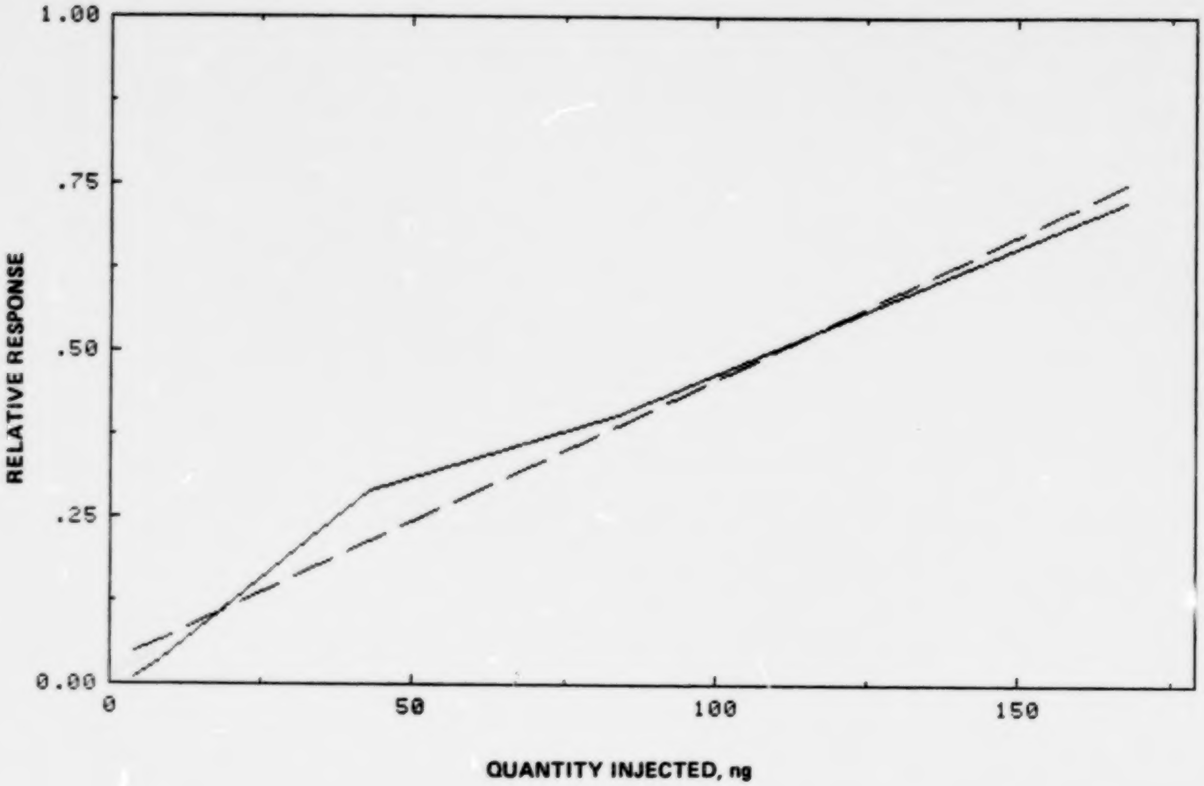
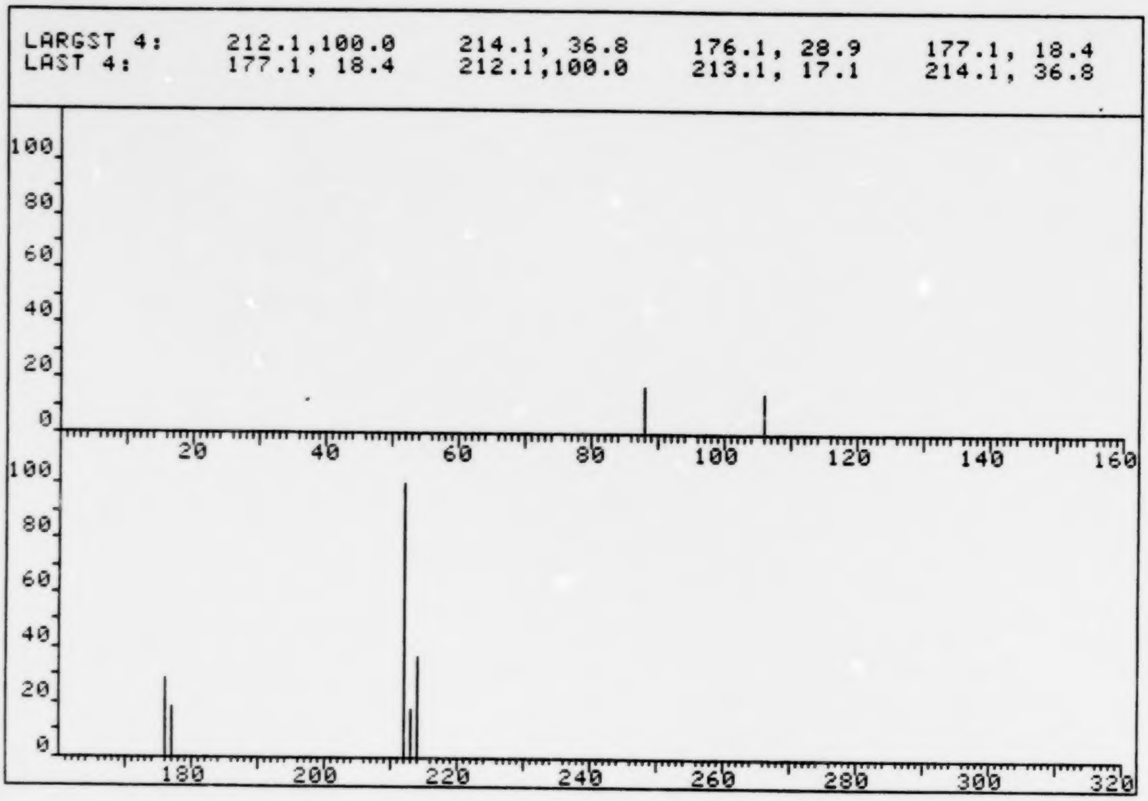


Figure B-65. Calibration curve for the determination of 1-amino-4-bromonaphthalene by GC/MS.

Range: 40 - 170 ng
Correlation coefficient: 0.987
Slope: 4.3×10^{-3}
y-Intercept: 3.0×10^{-2}



```
LARGST 4:    212.1,100.0    214.1, 36.8    176.1, 28.9    177.1, 18.4
LAST 4:      177.1, 18.4    212.1,100.0    213.1, 17.1    214.1, 36.8
```

Figure B-66. Mass spectrum for 2-chloroanthracene.

TABLE B-30. KEY IONS AND RELATIVE ABUNDANCES FOR 2-CHLOROANTHRACENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
88	17.1	176	28.9	212	100.0	214	36.8
106	14.5	177	18.4	213	17.1		

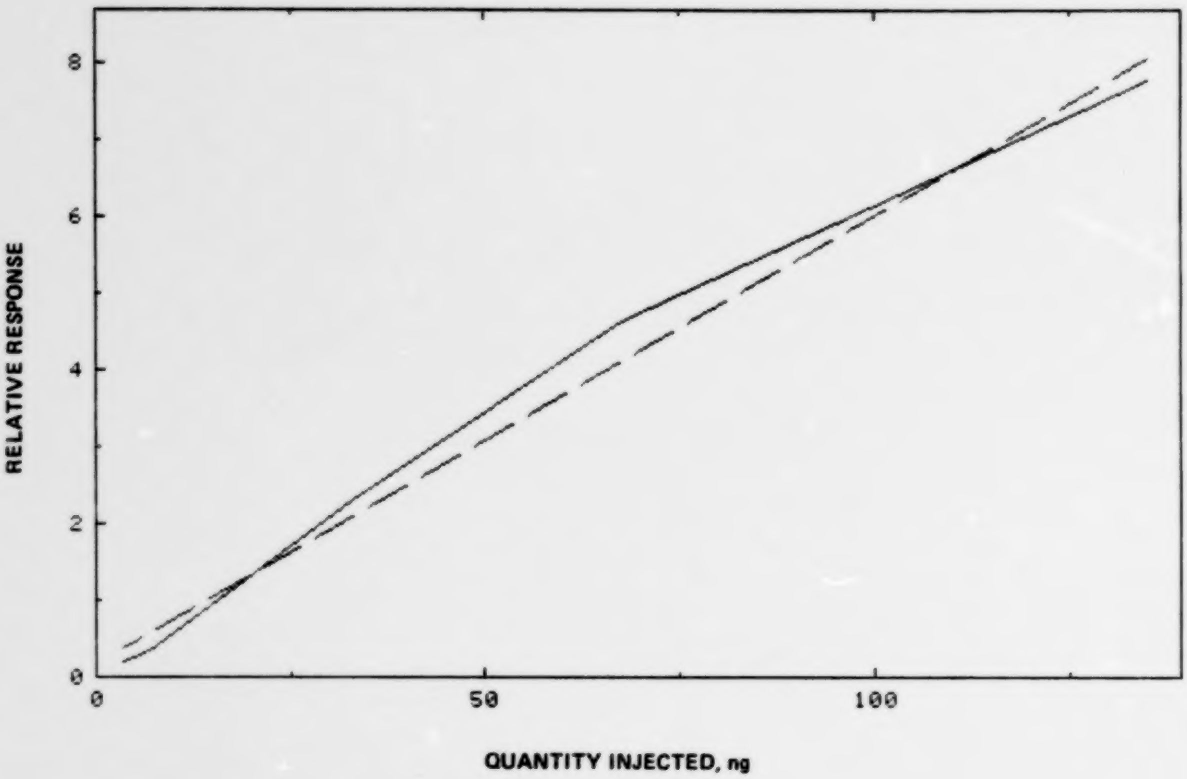
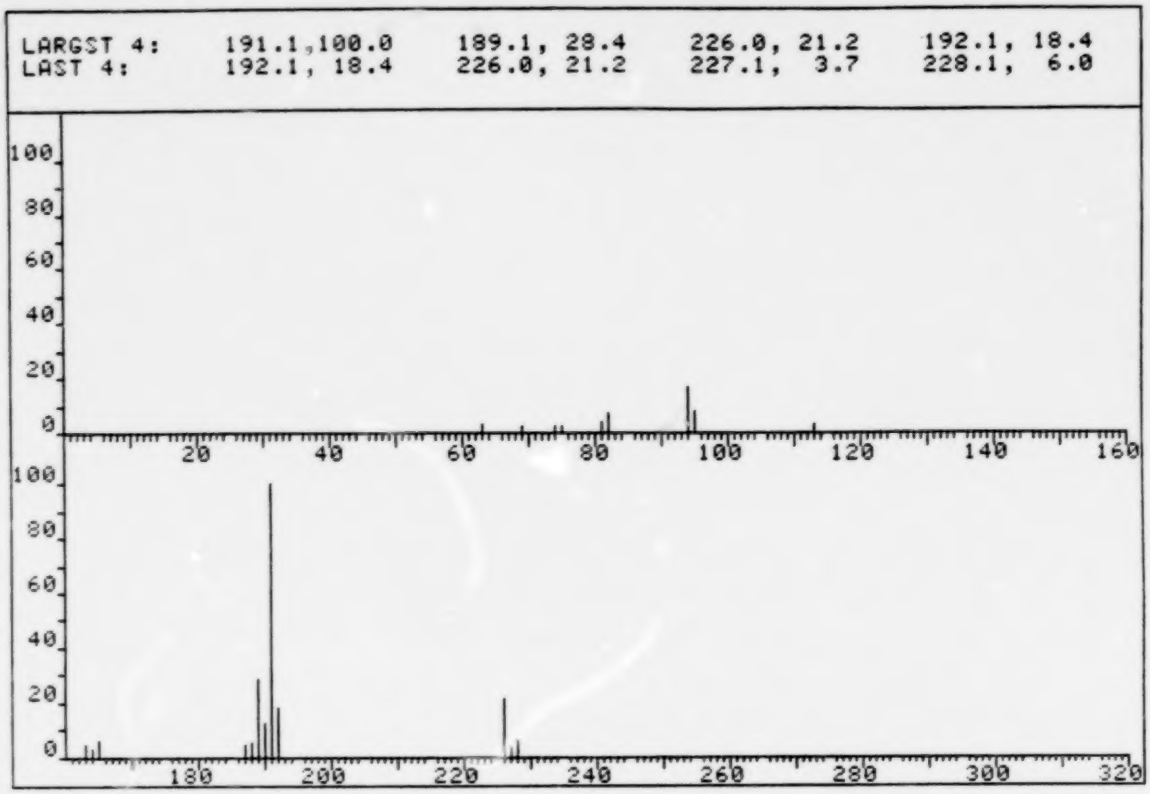


Figure B-67. Calibration curve for the determination of 2-chloroanthracene by GC/MS.

Range: 7.0 - 140 ng
Correlation coefficient: 0.994
Slope: 5.8×10^{-2}
y-Intercept: 1.8×10^{-1}



```
LARGST 4:     191.1,100.0    189.1, 28.4    226.0, 21.2    192.1, 18.4
LAST 4:       192.1, 18.4    226.0, 21.2    227.1,  3.7    228.1,  6.0
```

Figure B-68. Mass spectrum for 9-(chloromethyl)anthracene.

TABLE B-31. KEY IONS AND RELATIVE ABUNDANCES FOR 9-(CHLOROMETHYL)ANTHRACENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
63	3.0	94	16.7	187	4.9	226	21.2
69	2.3	95	7.9	188	5.1	227	3.7
74	2.6	113	3.5	189	28.4	228	6.0
75	2.6	163	4.9	190	13.0		
81	4.2	164	2.6	191	100.0		
82	7.4	165	5.8	192	18.4		

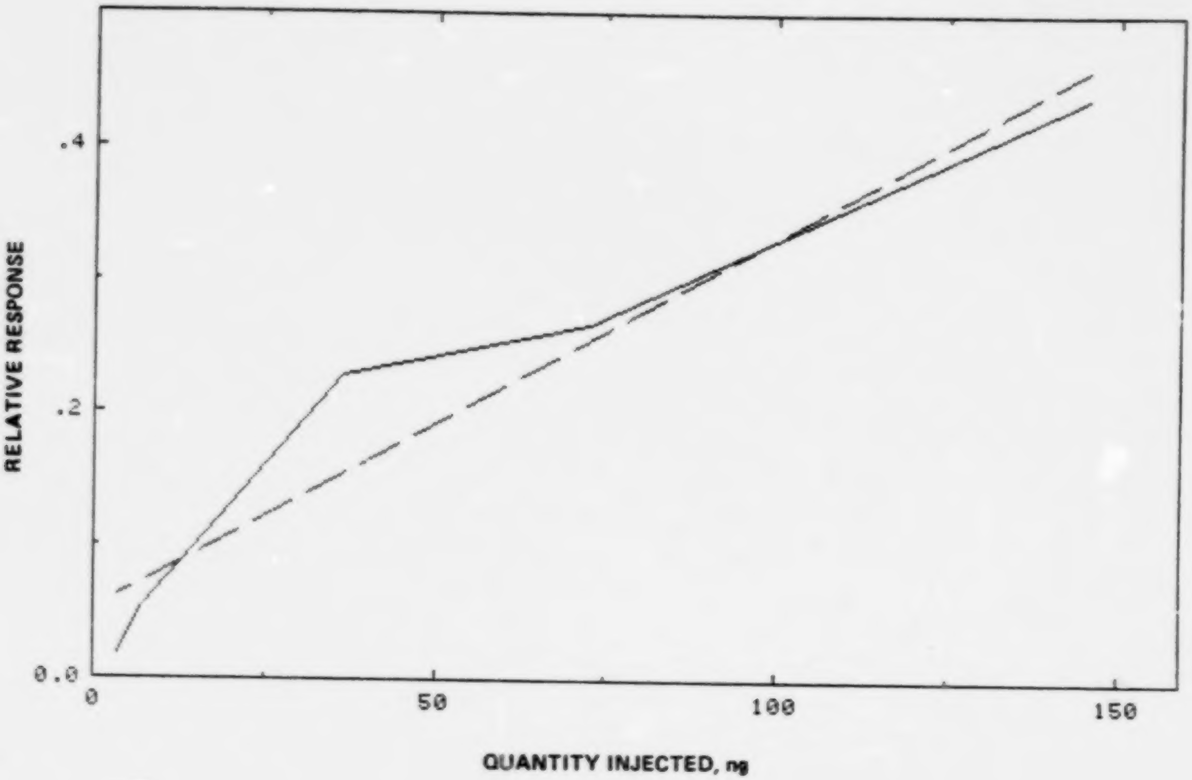
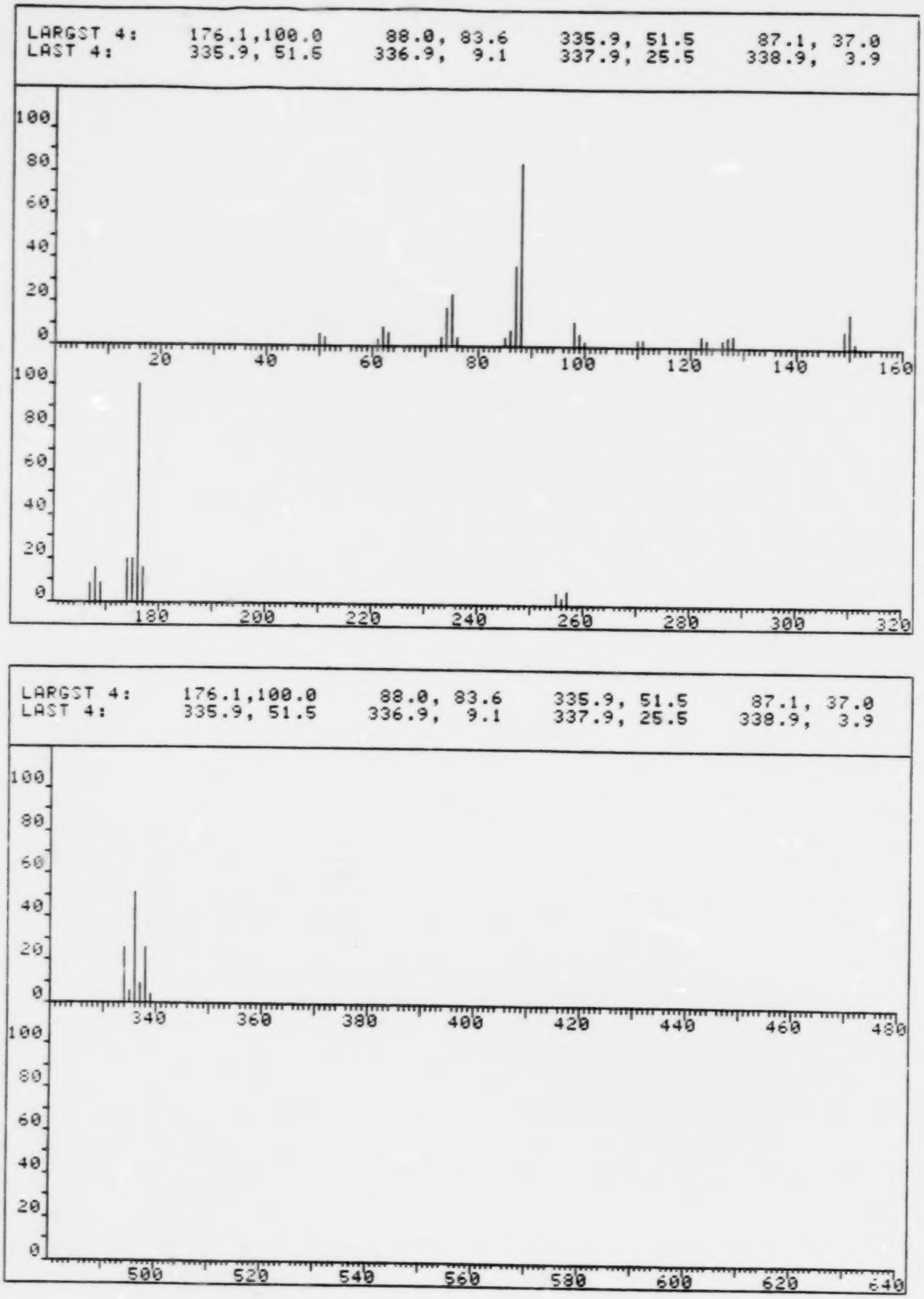


Figure B-69. Calibration curve for the determination of 9-(chloromethyl)anthracene by GC/MS.

Range: 7.0 - 150 ng
Correlation coefficient: 0.962
Slope: 2.8×10^{-3}
y-Intercept: 5.3×10^{-2}



```
LARGST 4:     176.1,100.0      88.0, 83.6     335.9, 51.5      87.1, 37.0
LAST 4:       335.9, 51.5     336.9,  9.1     337.9, 25.5     338.9,  3.9
```

```
LARGST 4:     176.1,100.0      88.0, 83.6     335.9, 51.5      87.1, 37.0
LAST 4:       335.9, 51.5     336.9,  9.1     337.9, 25.5     338.9,  3.9
```

Figure B-70. Mass spectrum for 9,10-dibromoanthracene.

TABLE B-32. KEY IONS AND RELATIVE ABUNDANCES FOR 9,10-DIBROMOANTHRACENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
50	5.6	87	37.0	128	5.6	255	5.6
51	4.4	88	83.6	149	8.1	256	3.2
61	3.4	98	11.5	150	15.9	257	5.9
62	9.1	99	5.6	151	2.7	334	25.7
63	6.6	100	2.4	167	8.8	335	5.4
73	4.2	110	3.7	168	15.7	336	51.5
74	17.6	111	3.7	169	8.6	337	9.1
75	24.0	122	5.1	174	19.9	338	25.5
76	4.2	123	3.7	175	20.1	339	3.9
85	3.9	126	3.7	176	100.0		
86	6.9	127	4.9	177	15.7		

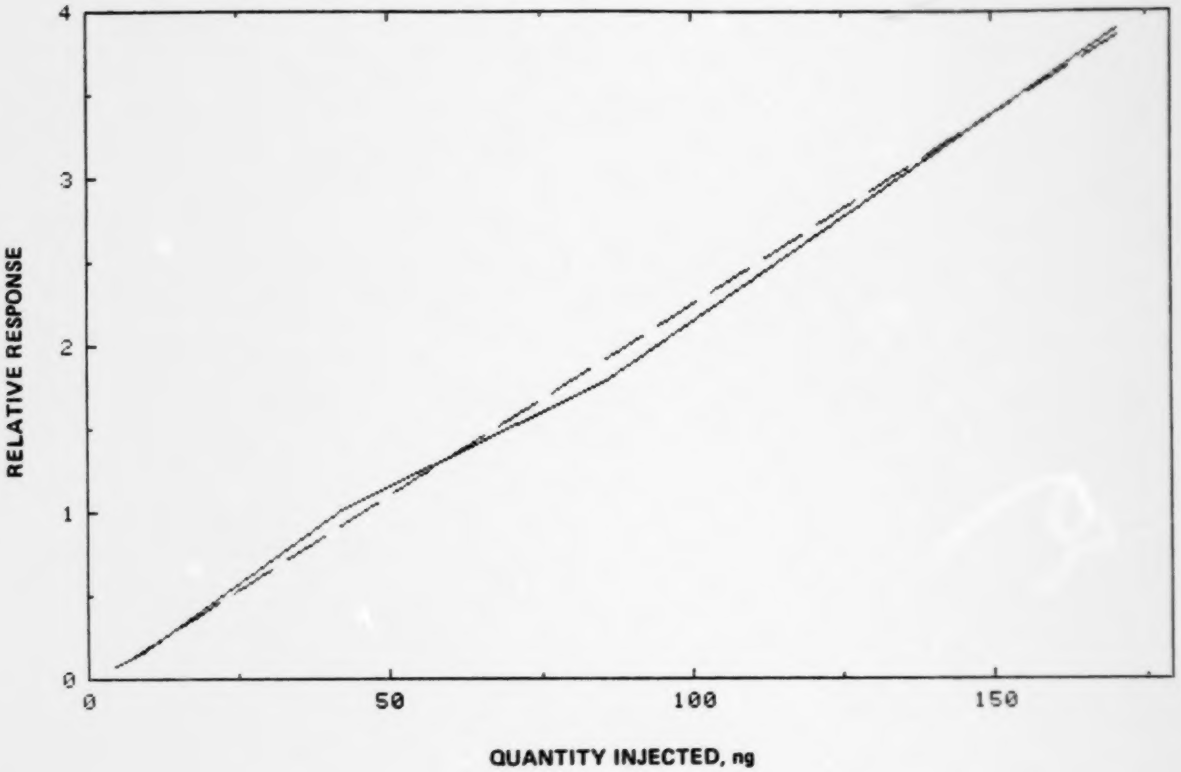


Figure B-71. Calibration curve for the determination of 9,10-dibromoanthracene by GC/MS.

Range: 4.0 - 170 ng
Correlation coefficient: 0.999
Slope: 2.3 x 10^{-2}
y-Intercept: -3.6 x 10^{-2}

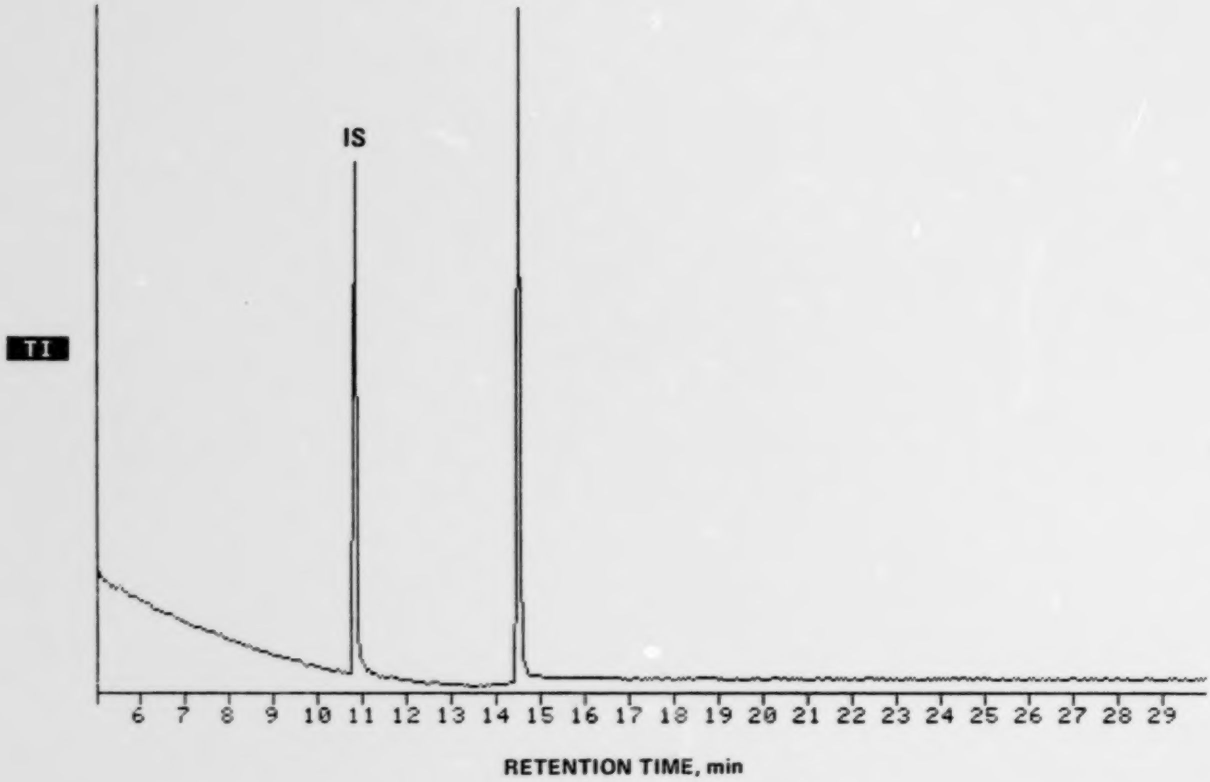
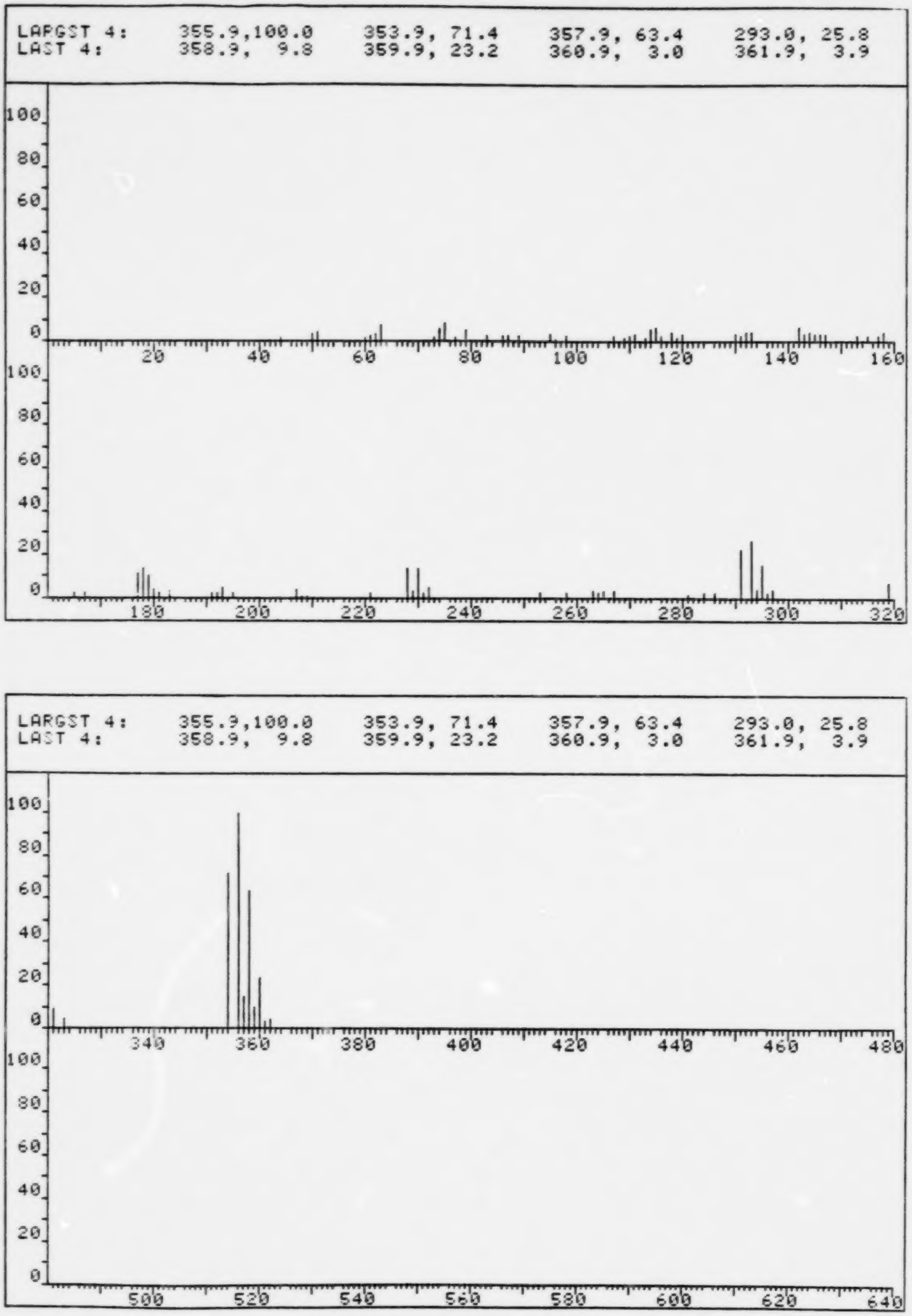


Figure B-72. Chromatogram by GC/MS for 1,2,3,4,7-pentachlorodibenzo-p-dioxin (t_R = 14.6 min).

Temperature program:
60 °C, 3 min isothermal
60 to 300 °C at 20 °C/min
300 °C, 10 min isothermal



```
LARGST 4:     355.9,100.0     353.9, 71.4     357.9, 63.4     293.0, 25.8
LAST 4:       358.9,   9.8    359.9, 23.2     360.9,   3.0    361.9,   3.9
```

```
LARGST 4:     355.9,100.0     353.9, 71.4     357.9, 63.4     293.0, 25.8
LAST 4:       358.9,   9.8    359.9, 23.2     360.9,   3.0    361.9,   3.9
```

Figure B-73. Mass spectrum for 1,2,3,4,7-pentachlorodibenzo-p-dioxin.

TABLE B-33. KEY IONS AND RELATIVE ABUNDANCES FOR 1,2,3,4,7-PENTACHLORODIBENZO-*p*-DIOXIN

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
47	2.2	110	8.0	169	3.0	242	1.0
48	1.1	111	12.0	171	.9	249	1.2
49	2.5	112	3.2	177	27.0	251	1.0
50	11.5	113	5.2	178	37.7	255	.8
51	14.8	114	21.1	179	28.5	256	3.1
52	1.1	115	18.0	180	9.2	257	1.2
53	3.3	116	6.6	181	7.4	258	3.2
60	5.5	118	13.9	183	5.1	260	1.1
61	5.0	119	3.3	184	.8	262	2.2
62	8.5	120	10.6	185	1.6	263	5.5
63	21.5	121	2.3	188	2.6	264	3.0
64	1.4	122	6.0	190	2.6	265	7.2
65	.8	123	2.6	191	.5	266	2.0
66	.8	125	1.1	192	4.5	267	3.8
71	4.2	126	2.5	193	8.1	268	.8
72	3.4	128	2.8	194	3.1	269	1.1
73	6.0	129	1.8	195	5.5	275	1.0
74	20.7	130	8.9	196	1.1	277	1.3
75	25.5	131	6.0	197	1.1	279	.9
76	2.9	132	13.9	200	.8	284	2.8
77	1.7	133	6.1	202	.9	285	1.1
79	20.9	134	3.6	204	1.1	286	2.9
80	1.7	135	1.6	205	1.1	288	1.3
83	5.4	138	2.4	207	.8	291	34.4
84	5.4	139	.9	209	2.0	293	40.6
85	5.1	140	1.0	211	1.6	294	6.1
86	6.7	142	26.3	212	1.9	295	21.1
87	9.1	143	11.4	213	1.1	296	3.2
88	3.0	144	14.2	214	2.2	297	5.0
89	3.5	145	9.3	215	.9	298	1.1
90	.8	146	10.7	216	1.4	319	7.6
93	.8	147	5.5	218	1.0	320	1.7
95	10.3	149	2.6	221	5.0	321	10.9
96	6.0	150	3.3	222	.9	322	1.6
97	8.6	151	2.0	223	3.9	323	4.8
98	7.9	153	7.7	225	.9	324	1.0
99	1.6	155	7.4	228	31.3	325	1.6
100	1.4	156	1.7	229	5.5	354	71.9
101	.9	157	6.1	230	31.4	356	100.0
102	1.3	158	8.6	231	3.8	357	14.8
103	.8	159	4.1	232	11.1	358	62.3
106	2.2	160	5.3	233	1.2	359	9.2
107	8.9	165	6.5	234	1.3	360	22.5
108	5.5	167	7.9	239	.8	361	3.0
109	7.0	168	.9	240	1.1	362	3.6

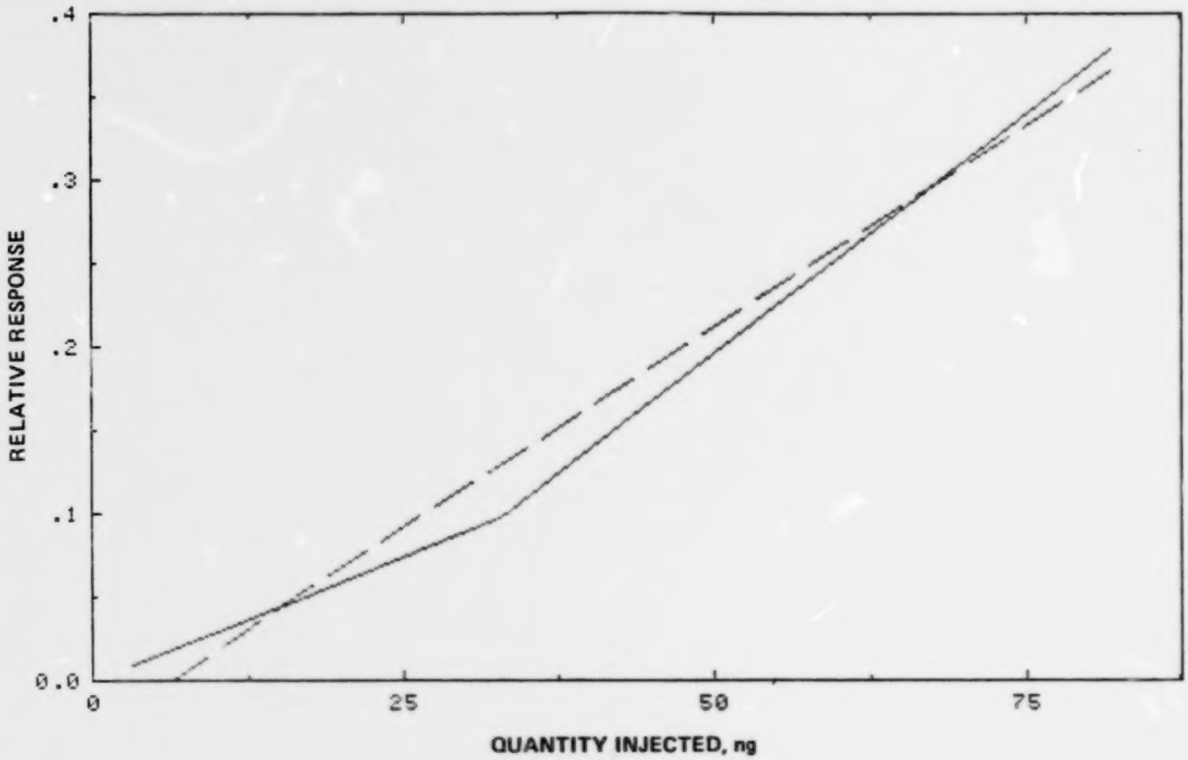


Figure B-74. Calibration curve for the determination of 1,2,3,4,7-pentachlorodibenzo-p-dioxin by GC/MS.

Range: 10 - 80 ng
Correlation coefficient: 0.990
Slope: 4.8×10^{-3}
y-Intercept: -2.8×10^{-2}

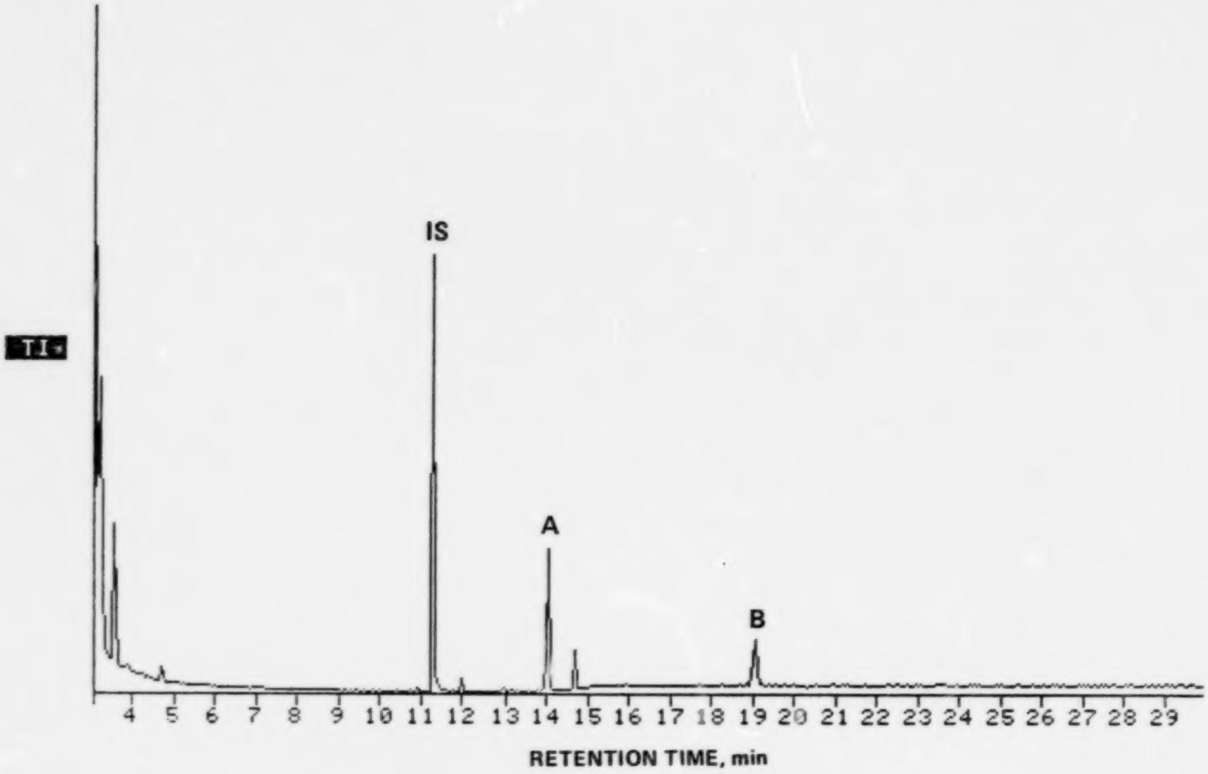
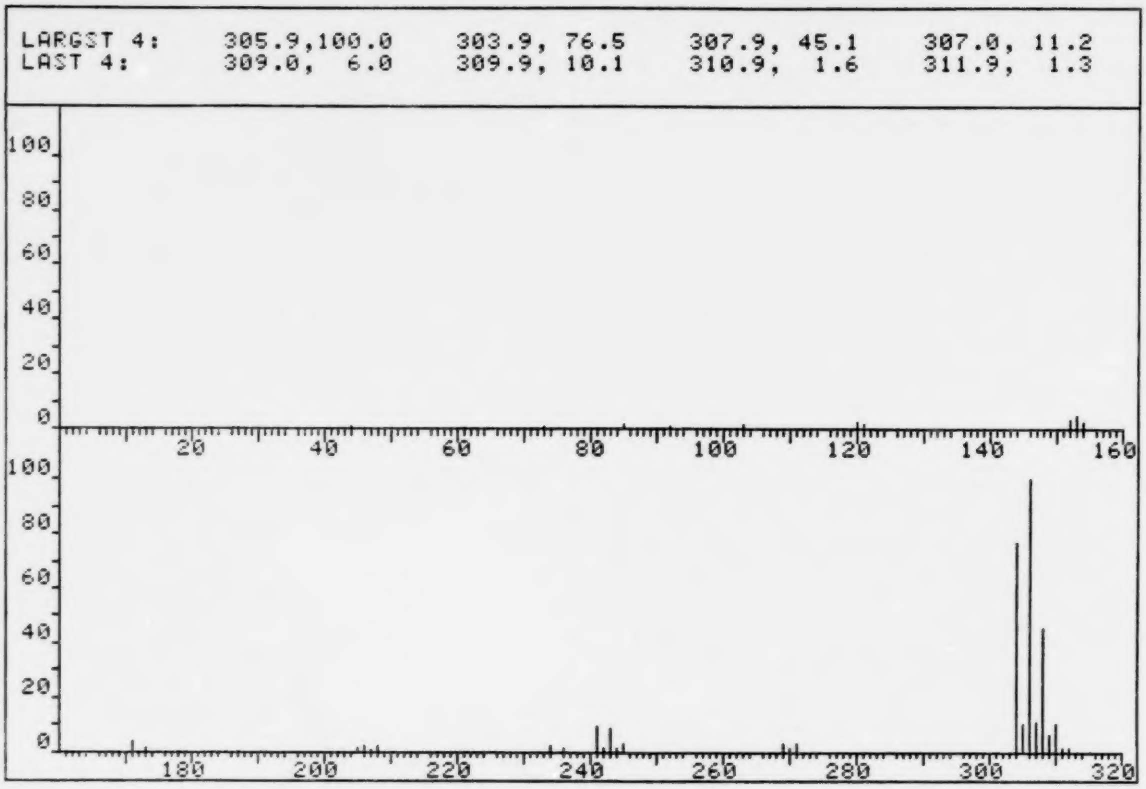


Figure B-75. Chromatogram by GC/MS for

(A) 2,3,7,8-tetrachlorodibenzofuran(t_R = 14.0 min),
(B) 1,2,3,4,6,7,8,9-octachlorodibenzo-p-dioxin
(t_R = 19.0 min), and
(C) 1,2,3,4,6,7,8,9-octachlorodibenzofuran(t_R = 19.1 min).

Temperature program:
60 °C, 3 min isothermal
60 to 300 °C at 20 °C/min
300 °C, 10 min isothermal



```
LARGST 4:    305.9,100.0    303.9, 76.5    307.9, 45.1    307.0, 11.2
LAST 4:      309.0,   6.0   309.9, 10.1    310.9,  1.6    311.9,  1.3
```

Figure B-76. Mass spectrum for 2,3,7,8-tetrachlorodibenzofuran.

TABLE B-34. KEY IONS AND RELATIVE ABUNDANCES FOR 2,3,7,8-TETRACHLORO-DIBENZOFURAN

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
73	1.2	173	1.4	243	8.8	307	11.2
85	2.0	205	1.6	244	1.3	308	45.1
103	1.7	206	2.0	245	2.9	309	6.0
120	2.3	207	.8	269	2.7	310	10.1
121	2.0	208	2.0	270	1.2	311	1.6
152	3.7	234	1.8	271	2.4	312	1.3
153	5.0	236	1.2	304	76.5		
154	2.5	241	9.2	305	10.4		
171	4.2	242	1.4	306	100.0		

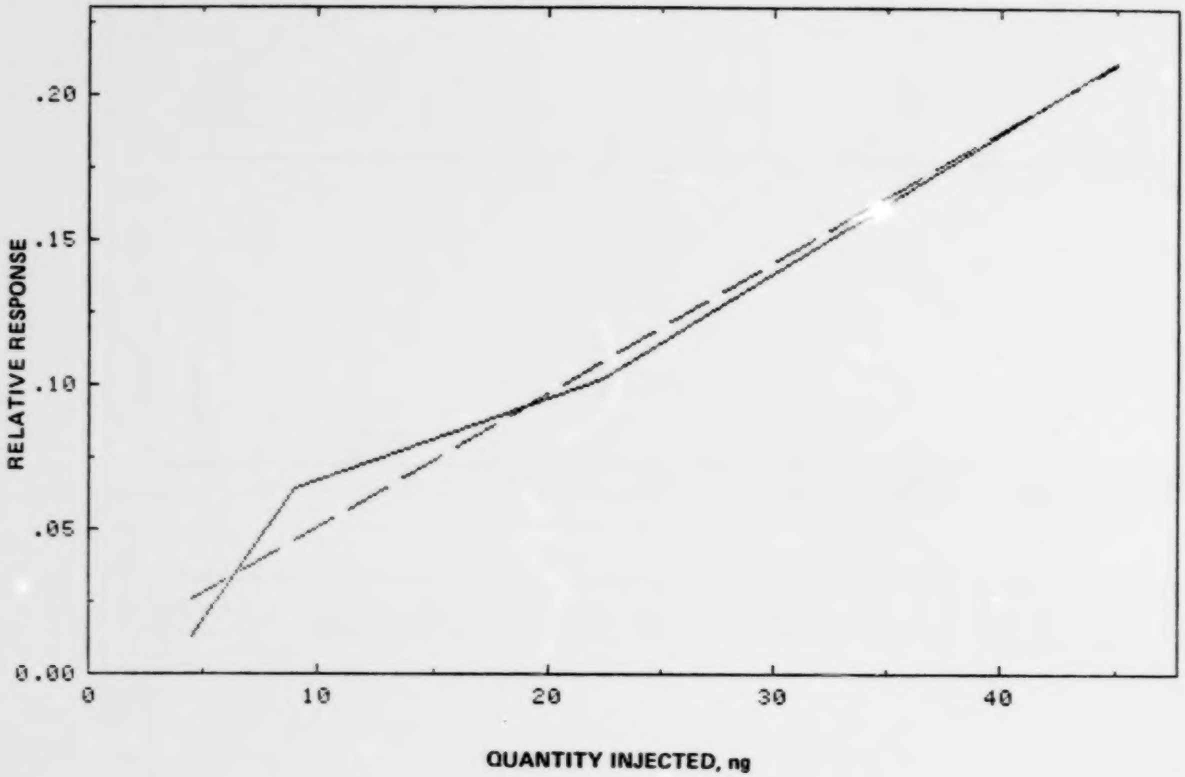
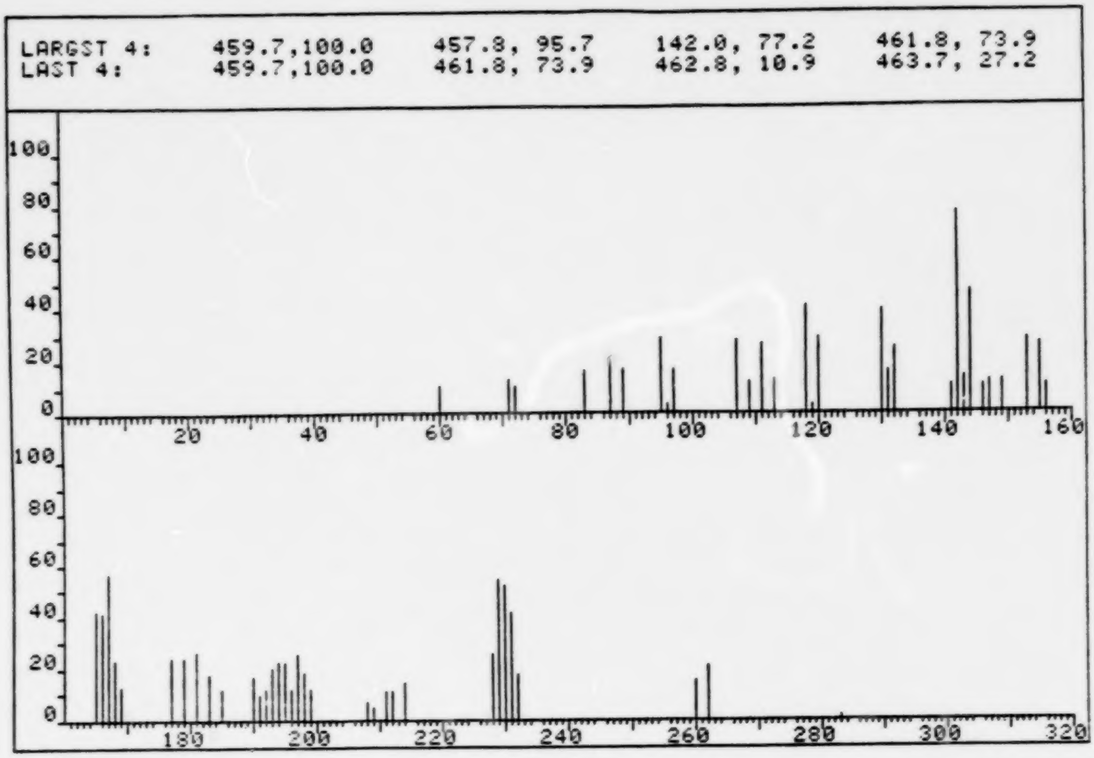


Figure B-77. Calibration curve for the determination of 2,3,7,8-tetrachlorodibenzofuran by GC/MS.

Range: 9.0 - 45 ng
Correlation coefficient: 0.987
Slope: 4.6×10^{-3}
y-Intercept: 5.1×10^{-3}



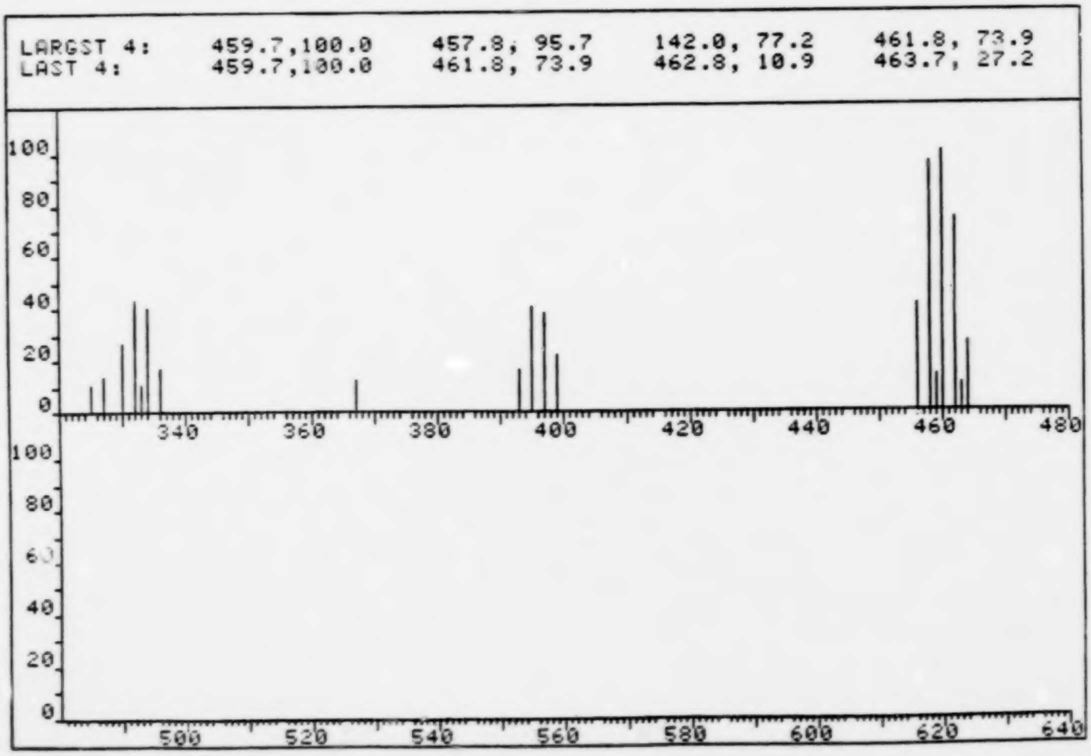


Figure B-78. Mass spectrum for 1,2,3,4,6,7,8,9-octachlorodibenzo-p-dioxin.

TABLE B-35. KEY IONS AND RELATIVE ABUNDANCES FOR 1,2,3,4,6,7,8,9-OCTACHLORO-DIBENZO-p-DIOXIN

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
60	10.9	143	14.1	193	19.6	327	14.1
71	13.0	144	46.7	194	21.7	330	27.2
72	10.9	146	10.9	195	21.7	332	43.5
83	16.3	147	12.0	196	12.0	333	10.9
87	21.7	149	12.0	197	25.0	334	40.2
89	17.4	153	28.3	198	18.5	336	17.4
95	29.3	155	27.2	199	12.0	367	12.0
96	3.3	156	10.9	208	6.5	393	16.3
97	17.4	165	42.4	209	4.3	395	40.2
107	28.3	166	41.3	211	10.9	397	38.0
109	12.0	167	56.5	212	10.9	399	21.7
111	27.2	168	22.8	214	14.1	456	41.3
113	13.0	169	13.0	228	25.0	458	95.7
118	41.3	177	23.9	229	54.3	459	14.1
119	3.3	179	23.9	230	52.2	460	100.0
120	29.3	181	26.1	231	41.3	462	73.9
130	39.1	183	17.4	232	17.4	463	10.9
131	16.3	185	12.0	260	15.2	464	27.2
132	25.0	190	16.3	262	20.7		
141	10.9	191	9.8	283	1.1		
142	77.2	192	12.0	325	10.9		

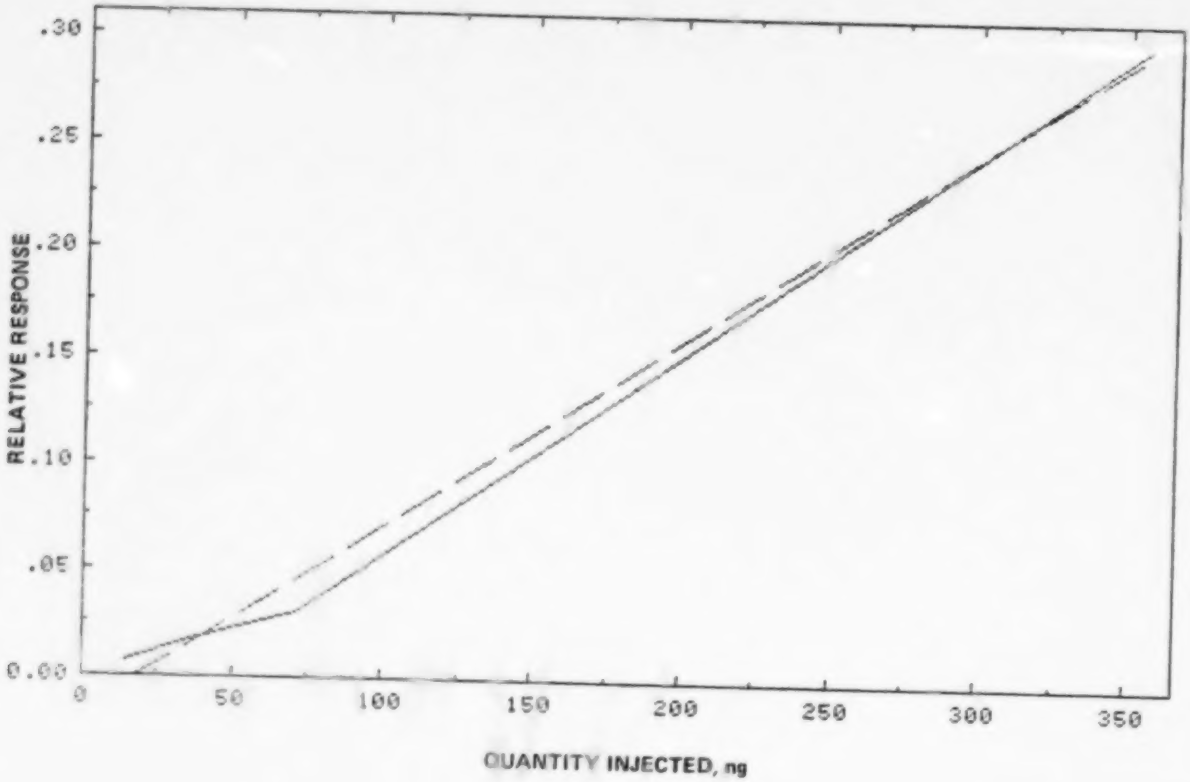
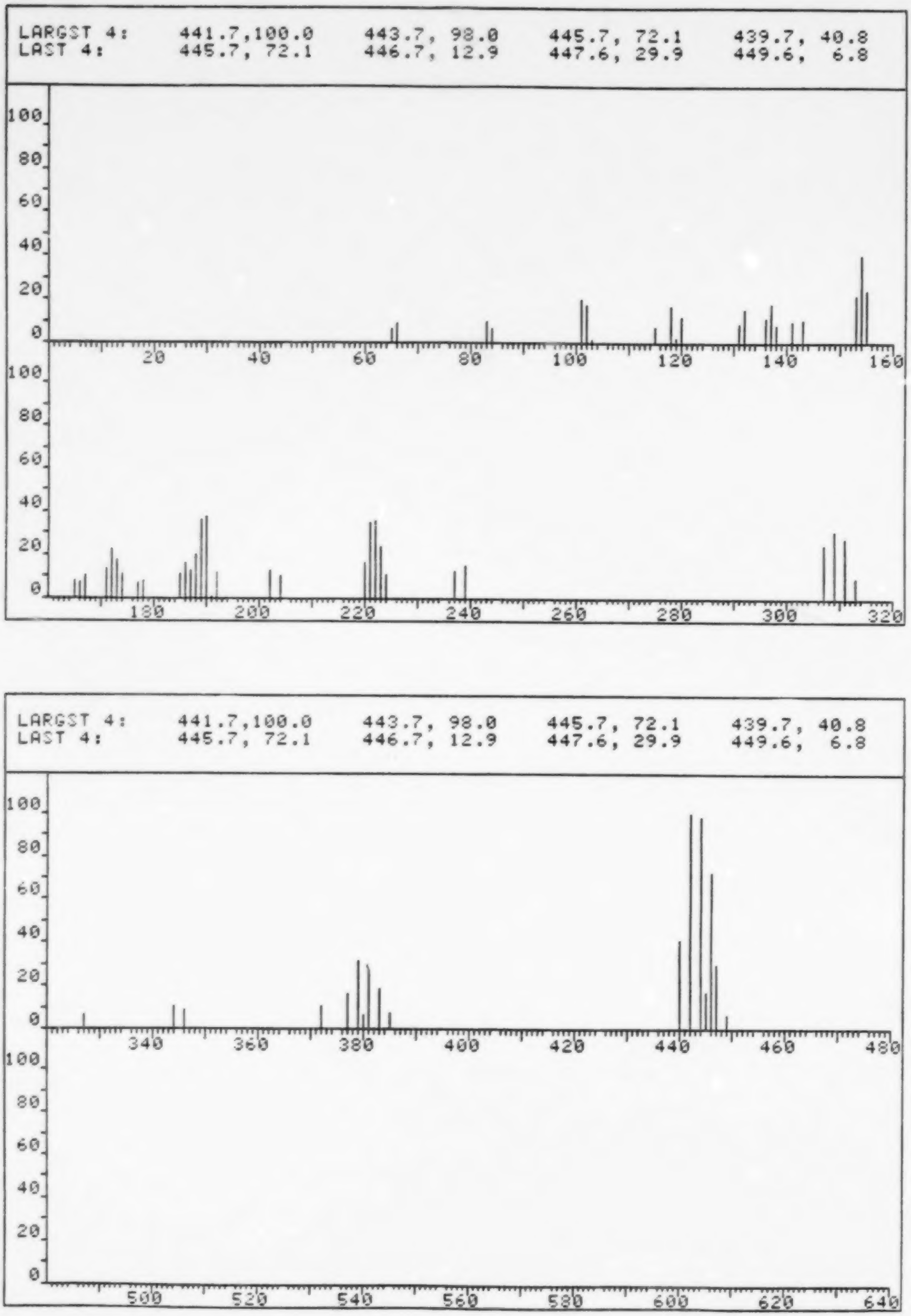


Figure B-79. Calibration curve for the determination of 1,2,3,4,6,7,8,9-octachlorodibenzo-p-dioxin by GC/MS.

Range: 40 - 360 ng
Correlation coefficient: 0.997
Slope: 8.8×10^{-4}
y-Intercept: -1.6×10^{-2}



```
LARGST 4:    441.7,100.0    443.7, 98.0    445.7, 72.1    439.7, 40.8
LAST 4:      445.7, 72.1    446.7, 12.9    447.6, 29.9    449.6,  6.8
```

```
LARGST 4:    441.7,100.0    443.7, 98.0    445.7, 72.1    439.7, 40.8
LAST 4:      445.7, 72.1    446.7, 12.9    447.6, 29.9    449.6,  6.8
```

Figure B-80. Mass spectrum for 1,2,3,4,6,7,8,9-octachlorodibenzofuran.

TABLE B-36. KEY IONS AND RELATIVE ABUNDANCES FOR 1,2,3,4,6,7,8,9-OCTACHLORODIBENZOFURAN

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	1.4	143	10.9	192	11.6	346	8.8
65	6.8	153	21.8	193	2.0	355	6.8
66	8.8	154	40.1	202	12.9	372	10.9
83	9.5	155	24.5	204	10.2	377	16.3
84	6.8	165	8.2	207	2.7	379	31.3
96	5.4	166	6.8	220	16.3	380	6.8
101	20.4	167	10.2	221	34.7	381	29.9
102	17.7	171	13.6	222	35.4	383	18.4
103	1.4	172	21.8	223	23.8	385	7.5
115	7.5	173	17.0	224	10.9	440	40.8
118	17.0	174	10.9	237	12.9	442	100.0
119	9.5	177	6.8	239	15.0	444	98.0
120	12.2	178	7.5	253	2.0	445	17.0
131	8.2	185	10.9	283	2.7	446	72.1
132	15.6	186	15.6	307	24.5	447	12.9
133	1.4	187	12.9	309	31.3	447	29.9
136	11.6	188	19.7	311	27.9	449	6.8
137	17.7	189	36.1	313	9.5		
138	8.2	190	37.4	327	6.8		
141	9.5	191	14.3	344	10.9		

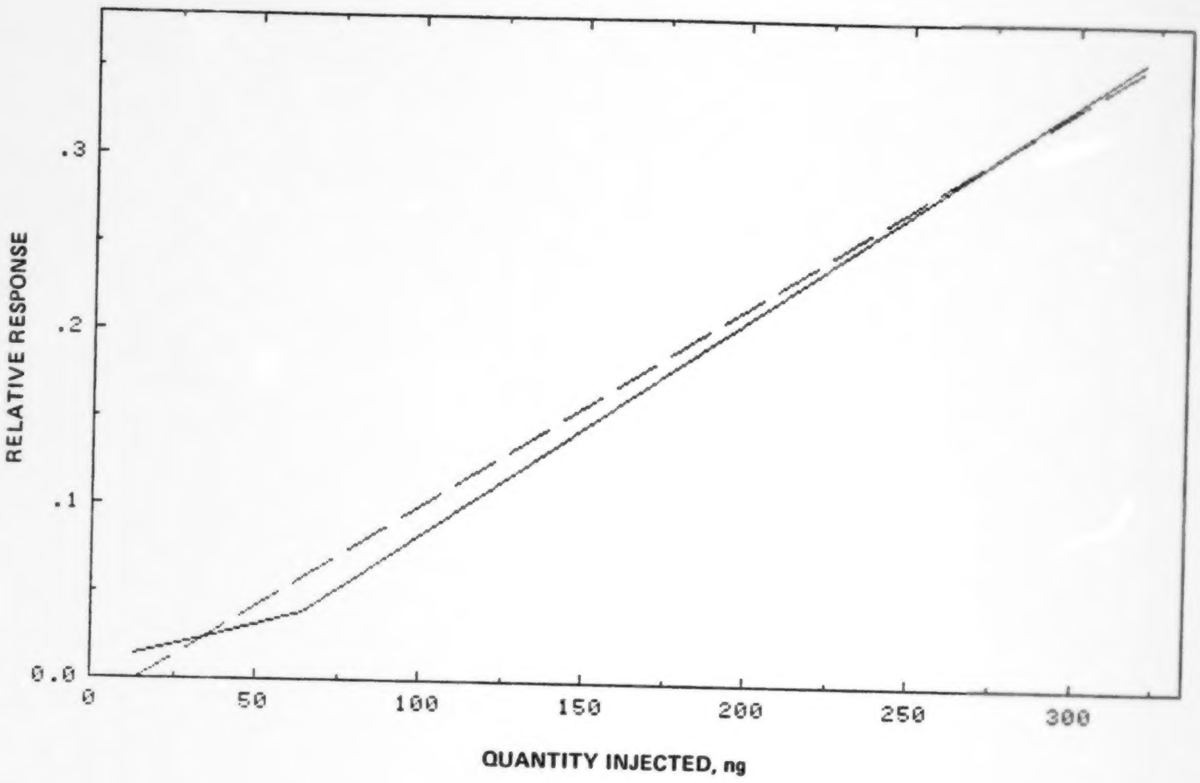


Figure B-81. Calibration curve for the determination of 1,2,3,4,6,7,8,9-octachlorodibenzofuran by GC/MS.

Range: 30 - 320 ng
Correlation coefficient: 0.996
Slope: 1.2×10^{-3}
y-Intercept: -1.6×10^{-2}

APPENDIX C

CHROMATOGRAMS AND CALIBRATION CURVES FOR GC/FID DETERMINATIONS

Index

Compound	Reference chromatogram Figure No.	Reference calibration curve Figure No.
3-Amino-9-ethyl carbazole	C-45	C-54
Aniline, 4,4'-methylenebis(*N*,*N*-dimethyl-	C-1	C-5
Aniline, *p*-(phenylazo)-	C-45	C-53
Aniline, 2,4,5-trimethyl-	C-32	C-35
o-Anisidine	C-32	C-34
o-Anisidine hydrochloride	C-19	C-22
o-Anisidine, 5-methyl-	C-45	C-50
o-Anisidine, 5-nitro-	C-32	C-36
Benzonitrile, 3,5-dibromo-4-hydroxy-	C-45	C-52
Biphenyl, 4-nitro	C-32	C-37
Carbamic acid, methyl- 2,3-dihydro-2,2-dimethyl-7-benzofuranyl ester	C-1	C-2
Carbamic acid, methyl-, 2,3-(dimethylmethylenedioxy)phenyl ester	C-7	C-8
Carbamic acid, methyl-, 1-naphthyl ester	C-1	C-3
Carbanilic acid, *m*-chloro-, 4-chloro-2-butynyl ester	C-7	C-10
Chlorobenzene	C-12	C-13
Coumarin, 3-chloro-7-hydroxy-4-methyl-, *O*-ester with *O*,*O*-diethyl phosphorothioate	C-25	C-31
Crotonic acid, 3-hydroxy-, *alpha*-methylbenzyl ester, dimethyl phosphate, (*E*)-	C-56	C-59
4-Cyclohexene-1,2-dicarboximide, *N*((1,1,2,2,-tetrachloroethyl)thio)-	C-7	C-11
4-Cyclohexene-1,2-dicarboximide, *N*-(trichloromethyl)thio-	C-1	C-4
m-Dichlorobenzene	C-14	C-15
o-Dichlorobenzene	C-16	C-18
p-Dichlorobenzene	C-16	C-17
Ether, 2,4-dichlorophenyl *p*-nitrophenyl	C-65	C-68
Heptachlor epoxide	C-65	C-67
Hexabromobiphenyl	C-1	C-6
Hydroquinone	C-19	C-23
Hydroxylamine, *N*-nitroso-*N*-phenyl-, ammonium salt	C-19	C-20
1,3,4-Metheno-1*H*-cyclobuta[cd]pentalene, 1,1a,2,2,3,3a,4,5,5,5a,5b,6-dodecachloro octahydro-	C-65	C-69

(continued)

APPENDIX C
(continued)

CHROMATOGRAMS AND CALIBRATION CURVES FOR GC/FID DETERMINATIONS

Index

Compound	Reference chromatogram Figure No.	Reference calibration curve Figure No.
1,4-Naphthoquinone, 2,3-dichloro-	C-62	C-64
2-Oxetanone (2-Oxepanone)	C-45	C-49
o-Phenylenediamine, 4-chloro-	C-19	C-24
Phosphonothioic acid, phenyl-, *O*-(4-bromo-2,5-dichloro-phenyl) *O*-methyl ester	C-25	C-30
Phosphoric acid, 2-chloro-1-(2,4-dichlorophenyl) vinyl diethyl ester	C-25	C-27
Phosphoric acid, 1,2-dibromo-2,2-dichloroethyl dimethyl ester	C-40	C-42
Phosphoric acid, dimethyl ester, ester with (*E*)-3-hydroxy-*N*,*N*-dimethylcrotonamide	C-25	C-26
Phosphoric acid, trimethyl ester	C-32	C-33
Phosphoric acid, tri-*o*-tolyl ester	C-32	C-39
Phosphoric triamide, hexamethyl-	C-40	C-41
Phosphorodithioic acid, *S*-(((*p*-chlorophenyl)thio)methyl) *O*,*O*-diethyl ester	C-25	C-28
Phosphorodithioic acid, *O*,*O*-dimethyl ester, *S*-ester with 3-(mercaptomethyl)1,2,3-benzotriazin-4(3*H*)-one	C-56	C-61
Phosphorodithioic acid, *O*,*O*-dimethyl ester, *S*-ester with *N*-(mercaptomethyl)phthalimide	C-25	C-29
Phosphorodithioic acid, *S*,*S*'-methylene *O*,*O*,*O*',*O*'-tetraethyl ester	C-56	C-60
Phosphorothioic acid, *O*,*O*-diethyl *O*-(*p*-(methylsulfinyl) phenyl) ester	C-40	C-44
Phosphorothioic acid, *O*,*O*-dimethyl-, *O*-(4-(methylthio)-*m*-tolyl) ester	C-40	C-43
Propionitrile, 2-hydroxy-	C-45	C-46
Pyridine, 3-chloromethyl-, hydrochloride	C-19	C-21
Pyridine, 2,6-diamino-3-(phenylazo)-, monohydrochloride	C-45	C-55
Styrene	C-45	C-47
Succinic acid, mercapto-, diethyl ester, *S*-ester with *O*,*O*-dimethyl phosphorodithioate	C-56	C-58

(continued)

APPENDIX C
(continued)

CHROMATOGRAMS AND CALIBRATION CURVES FOR GC/FID DETERMINATIONS

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Compound	Reference chromatogram Figure No.	Reference calibration curve Figure No.
Tetraethylpyrophosphate	C-56	C-57
Toluene, 2,4-diamino-	C-45	C-51
o-Toluidine	C-45	C-48
o-Toluidine, 5-chloro-	C-62	C-63
o-Toluidine, 4-(*o*-tolylazo)-	C-32	C-38
p-Toluidine, *alpha*,*alpha*,*alpha*-trifluoro-2,6-dinitro-*N*,*N*-dipropyl-	C-65	C-66
S-Triazine, 2,4-dichloro-6-(*o*-chloroanilino)-	C-7	C-9

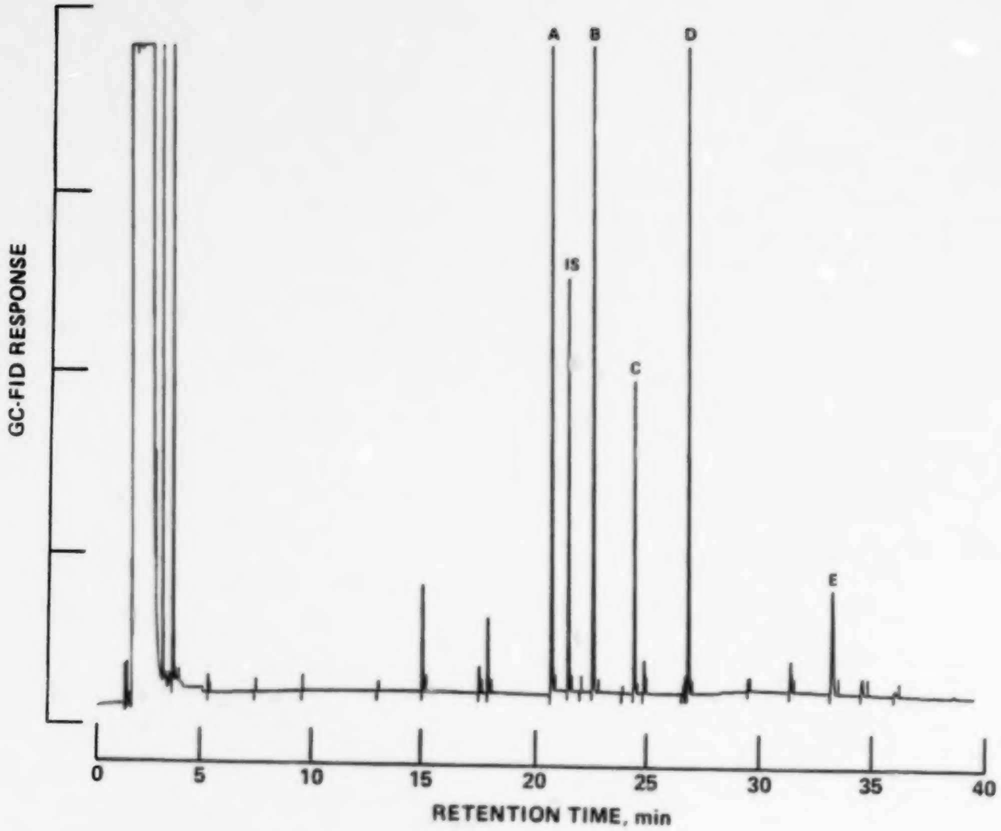

Figure C-1. Chromatogram by GC/FID for

(A) carbamic acid, methyl-, 2,3-dihydro-2,2-dimethyl-7-benzofuranyl ester(t_R = 20.9 min),
(B) carbamic acid, methyl-, 1-naphthyl ester(t_R = 22.8 min),
(C) 4-cyclohexene-1,2-dicarboximide, N-(trichloromethyl)thio-(t_R = 24.7 min),
(D) aniline, 4,4'-methylenebis(N,N-dimethyl- (t_R = 27.2 min), and
(E) hexabromobiphenyl(t_R = 33.8 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

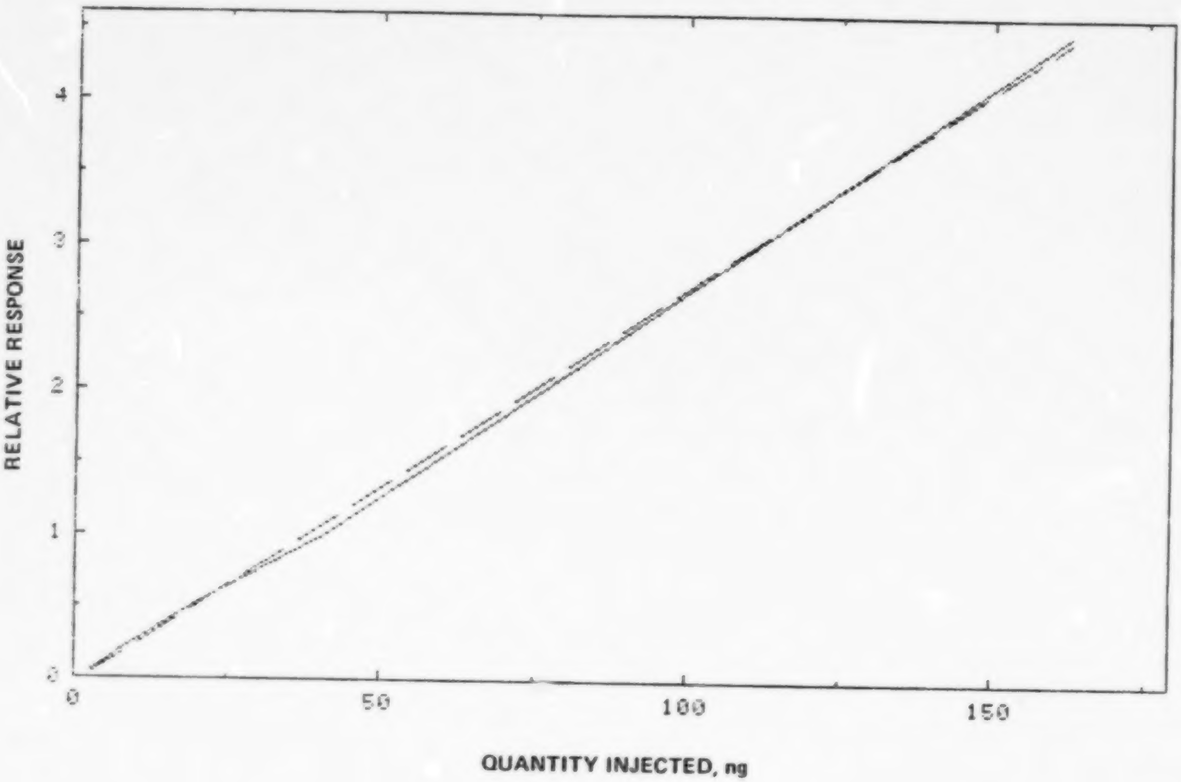

Figure C-2. Calibration curve for the determination of carbamic acid, methyl-, 2,3-dihydro-2,2-dimethyl-7-benzofuranyl ester by GC/FID.

Range: 8.1 - 160 ng
Correlation coefficient: 0.999
Slope: 2.8×10^{-2}
y-Intercept: -4.0×10^{-2}

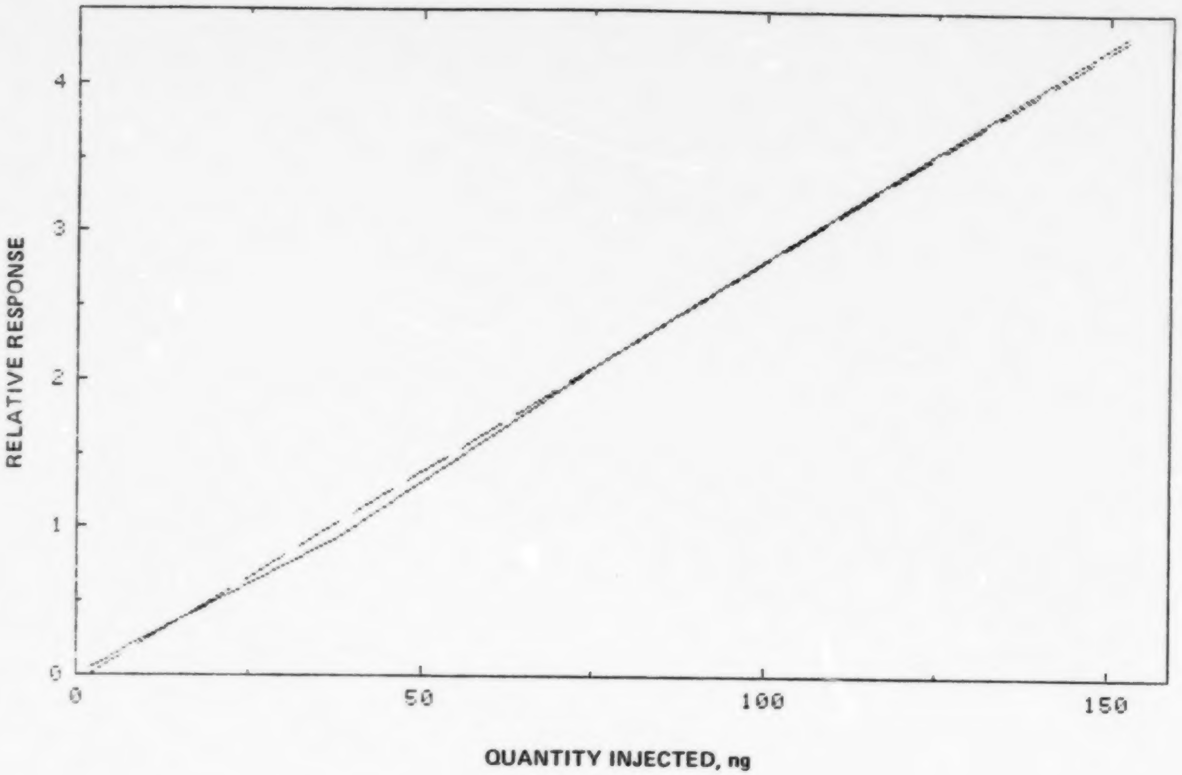

Figure C-3. Calibration curve for the determination of carbamic acid, methyl-, 1-naphthyl ester by GC/FID.

Range: 7.6 - 150 ng
Correlation coefficient: 0.999
Slope: 2.9×10^{-2}
y-Intercept: -5.4×10^{-2}

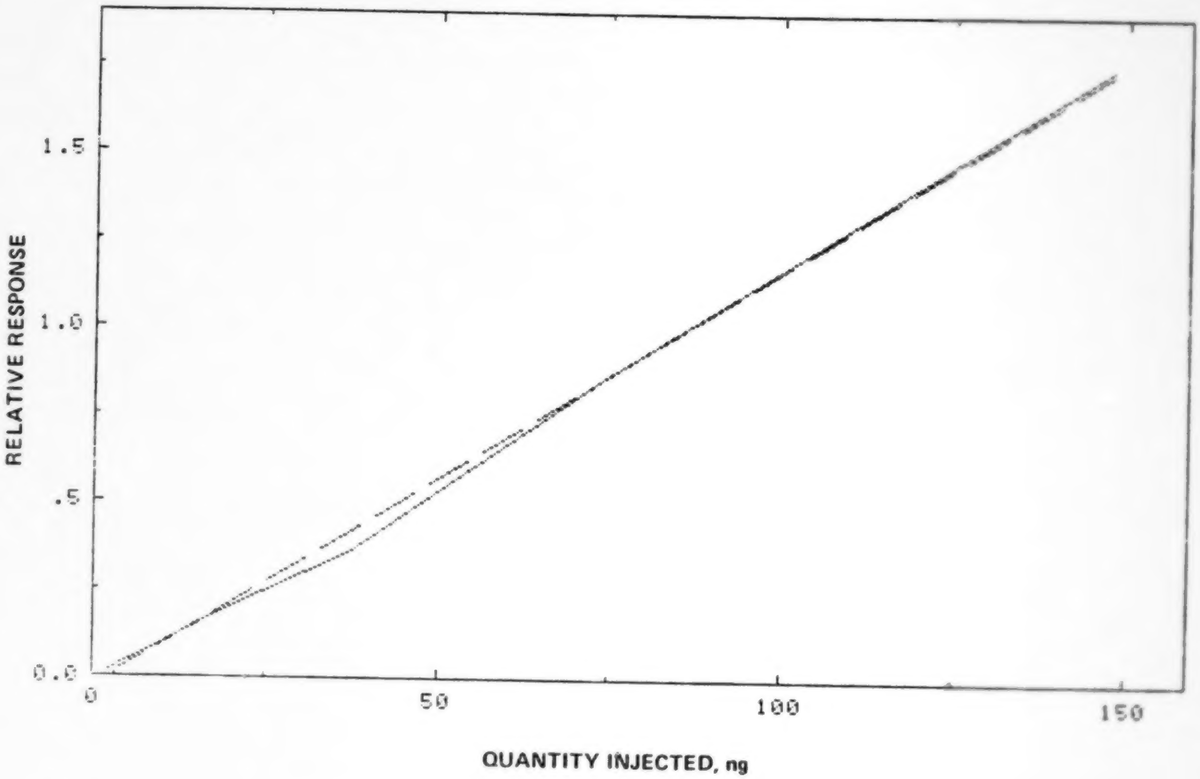

Figure C-4. Calibration curve for the determination of 4-cyclohexene-1,2-dicarboximide, N-(trichloromethyl)thio- by GC/FID.

Range: 7.4 - 150 ng
Correlation coefficient: 0.999
Slope: 1.2×10^{-2}
y-Intercept: -2.8×10^{-2}

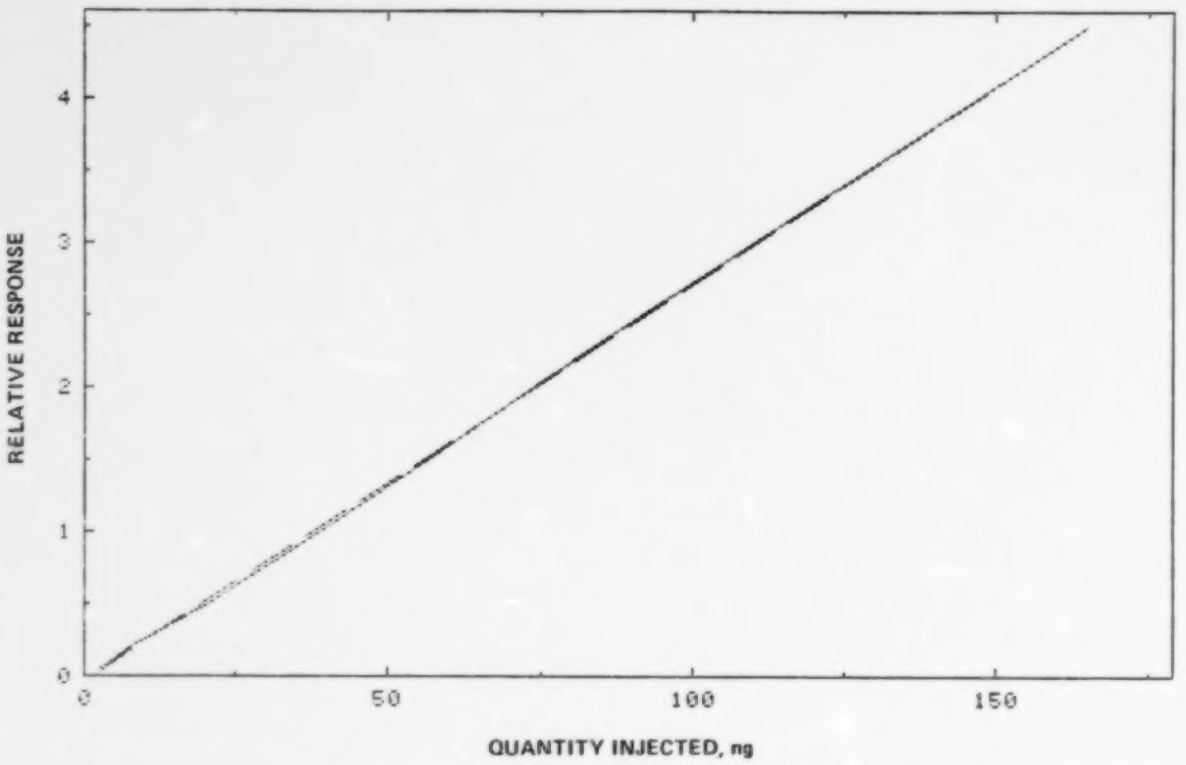

Figure C-5. Calibration curve for the determination of aniline, 4,4'-methylenebis(N,N-dimethyl- by GC/FID.

Range: 8.2 - 160 ng
Correlation coefficient: 0.999
Slope: 2.8×10^{-2}
y-Intercept: -3.6×10^{-2}

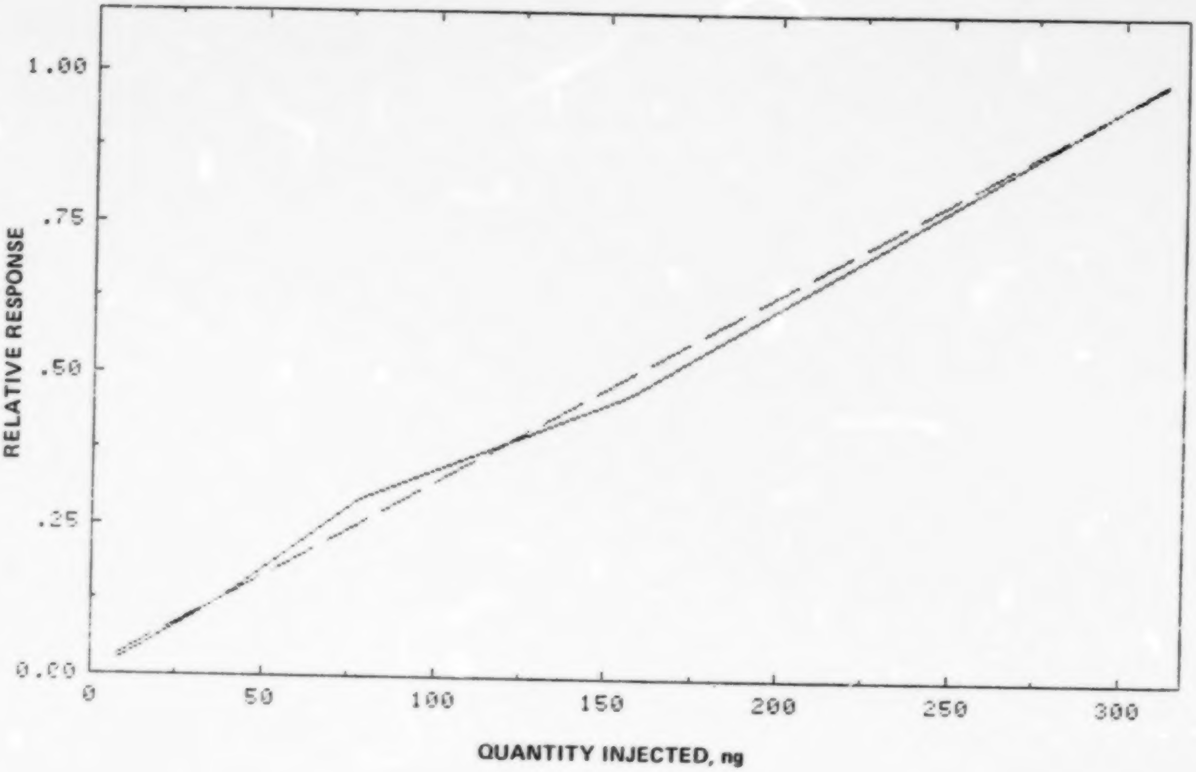

Figure C-6. Calibration curve for the determination of hexabromobiphenyl by GC/FID.

Range: 16 - 310 ng
Correlation coefficient: 0.998
Slope: 3.1×10^{-3}
y-Intercept: 8.1×10^{-3}

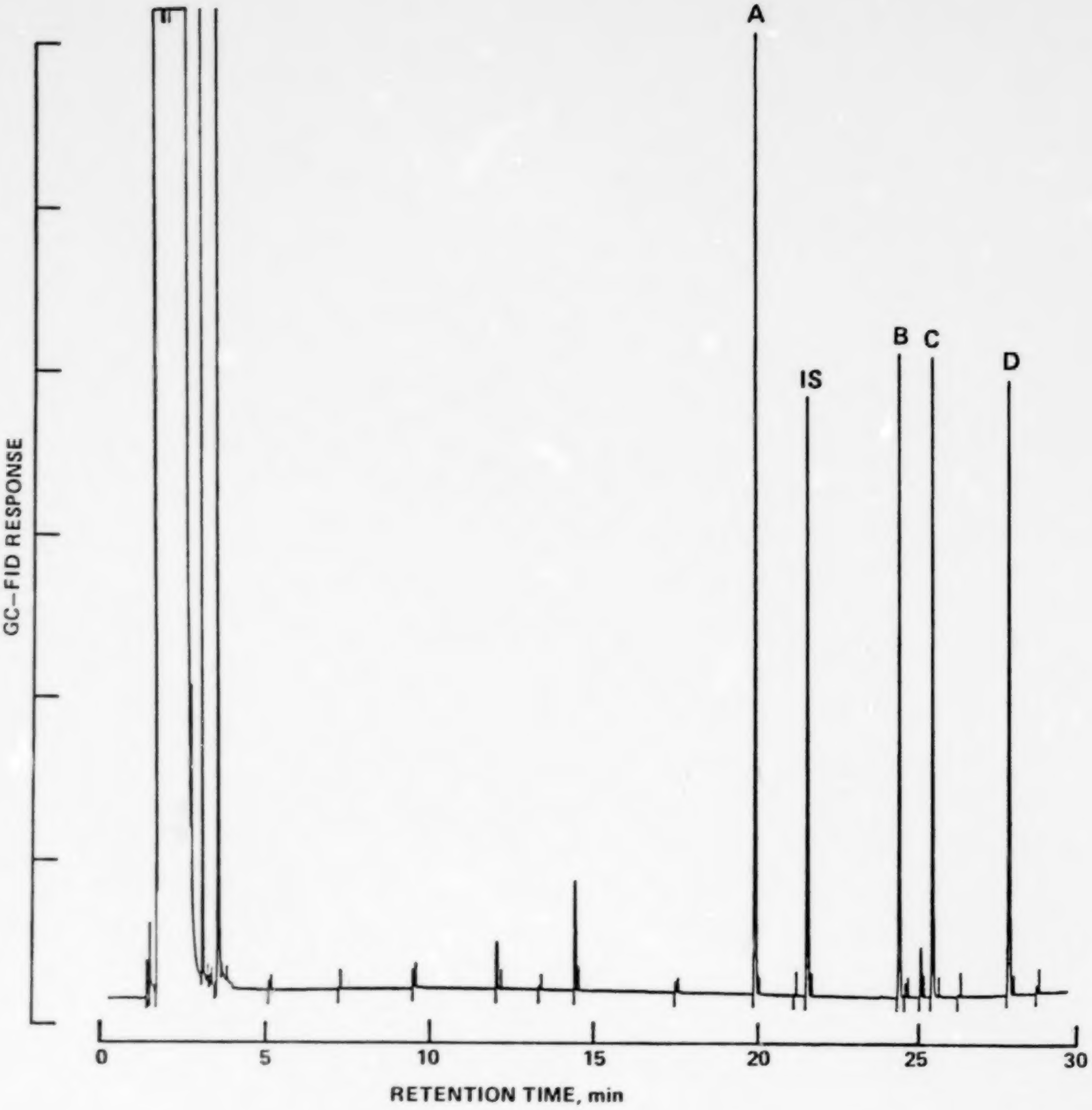

Figure C-7. Chromatogram by GC/FID for

(A) carbamic acid, methyl-, 2,3-(dimethylmethylenedioxy)phenyl ester(t_R = 20.0 min),
(B) S-triazine, 2,4-dichloro-6-(o-chloroanilino)-(t_R = 24.5 min),
(C) carbanilic acid, m-chloro-, 4-chloro-2-butynyl ester(t_R = 25.5 min), and
(D) 4-cyclohexene-1,2-dicarboximide, N-((1,1,2,2-tetrachloroethyl)thio)- (t_R = 27.8 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

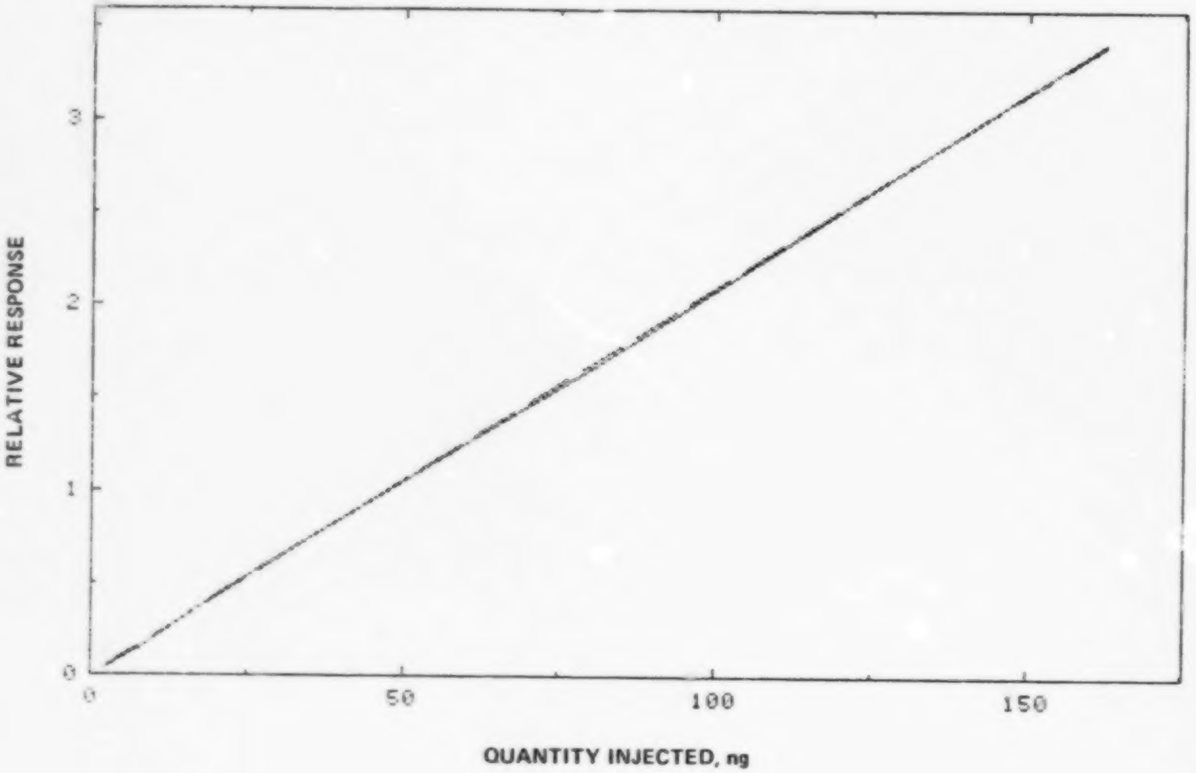

Figure C-8. Calibration curve for the determination of carbamic acid, methyl-, 2,3-(dimethylmethylenedioxy)phenyl ester by GC/FID.

Range: 2.0 - 160 ng
Correlation coefficient: 0.999
Slope: 2.1 x 10^{-2}
y-Intercept: -6.8 x 10^{-3}

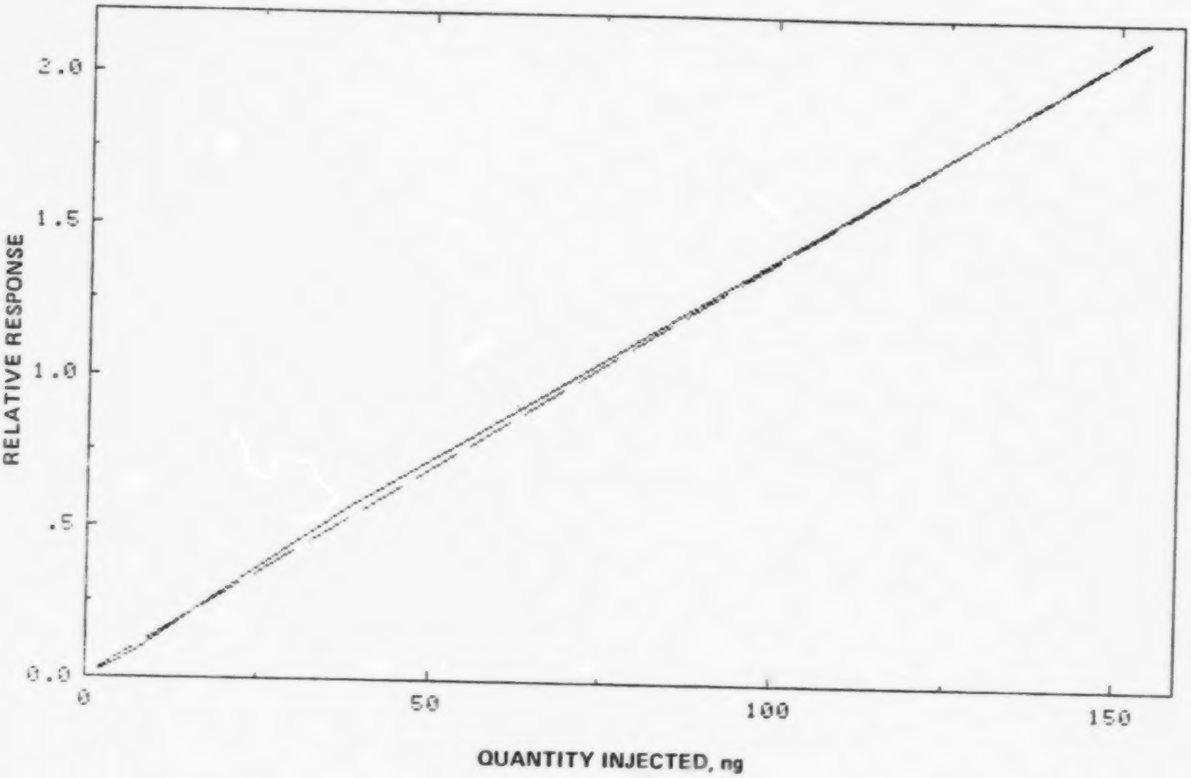

Figure C-9. Calibration curve for the determination of S-triazine, 2,4-dichloro-6-(o-chloroanilino)- by GC/FID.

Range: 1.9 - 150 ng
Correlation coefficient: 0.999
Slope: 1.4×10^{-2}
y-Intercept: 2.1×10^{-3}

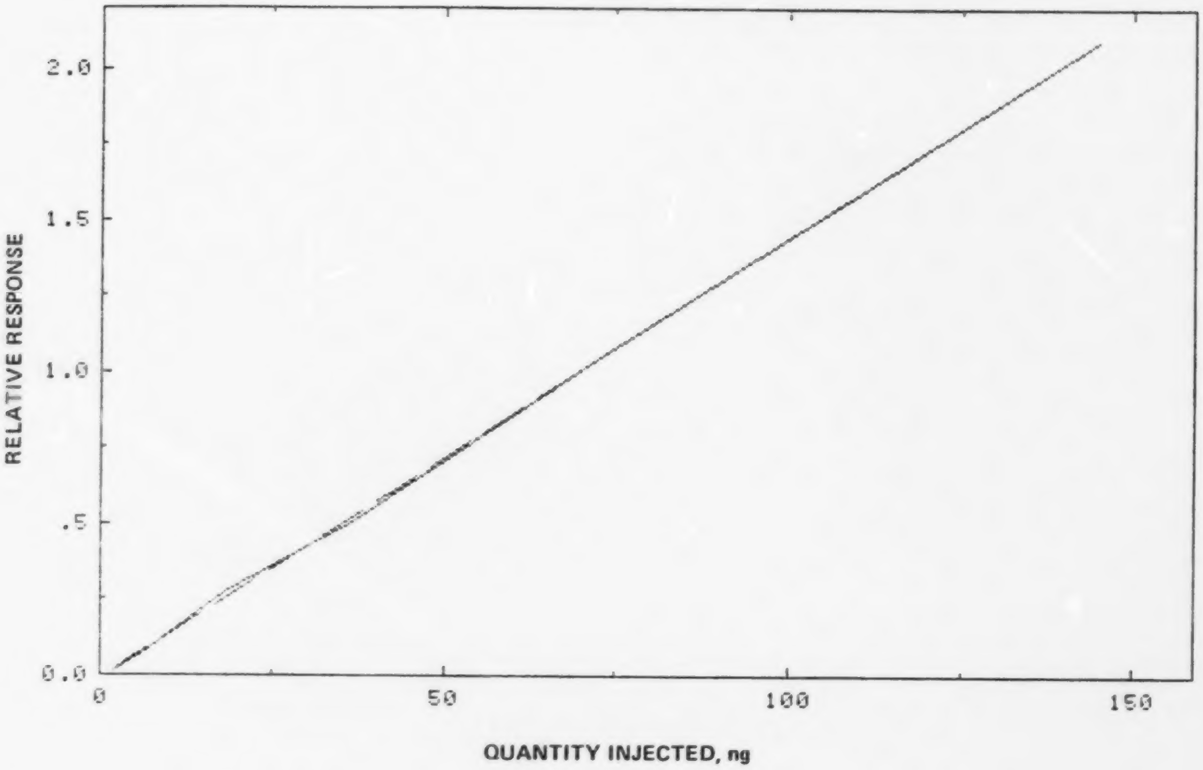

Figure C-10. Calibration curve for the determination of carbanilic acid, m-chloro-, 4-chloro-2-butynyl ester by GC/FID.

Range: 1.8 - 140 ng
Correlation coefficient: 0.999
Slope: 1.4×10^{-2}
y-Intercept: -1.1×10^{-2}

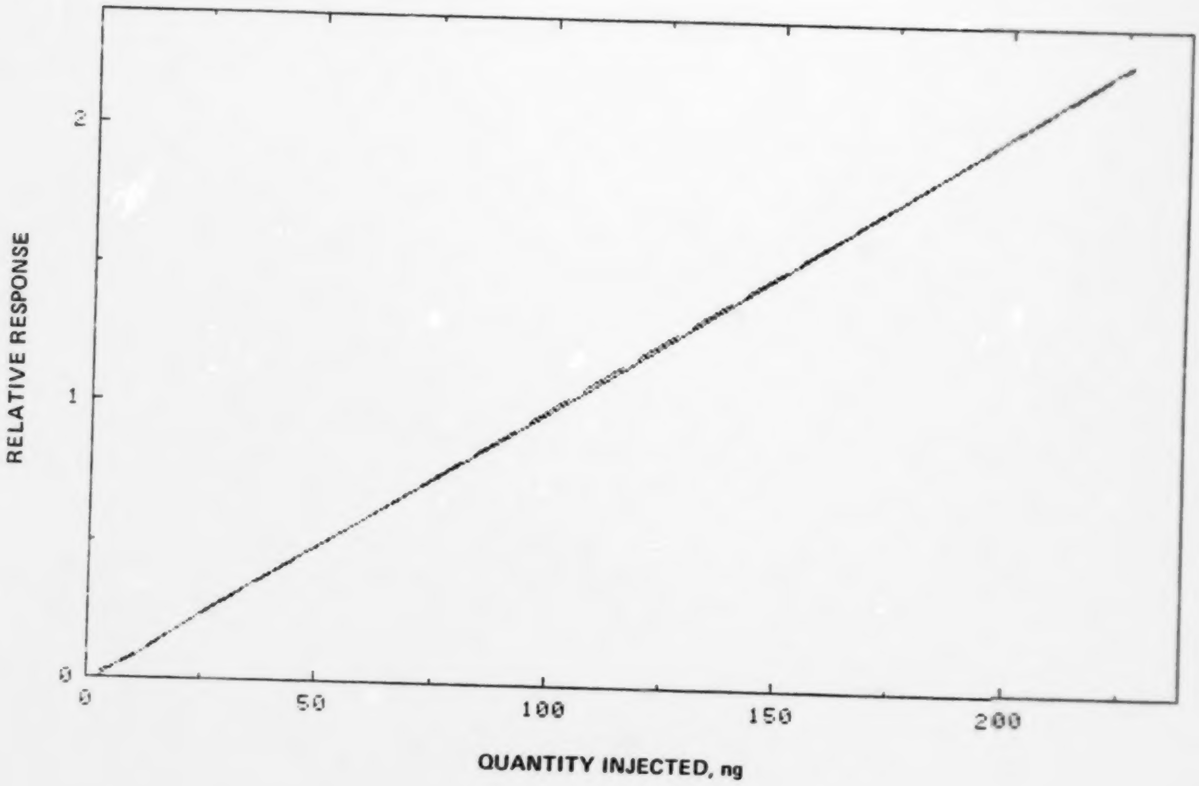

Figure C-11. Calibration curve for the determination of 4-cyclohexene-1,2-dicarboxemide, N-((1,1,2,2-tetrachloroethyl)thio)- by GC/FID.

Range: 5.7 - 2[illegible] ng
Correlation coefficient: 0.999
Slope: 1.0 x 10^{-2}
y-Intercept: -1.7 x 10^{-2}

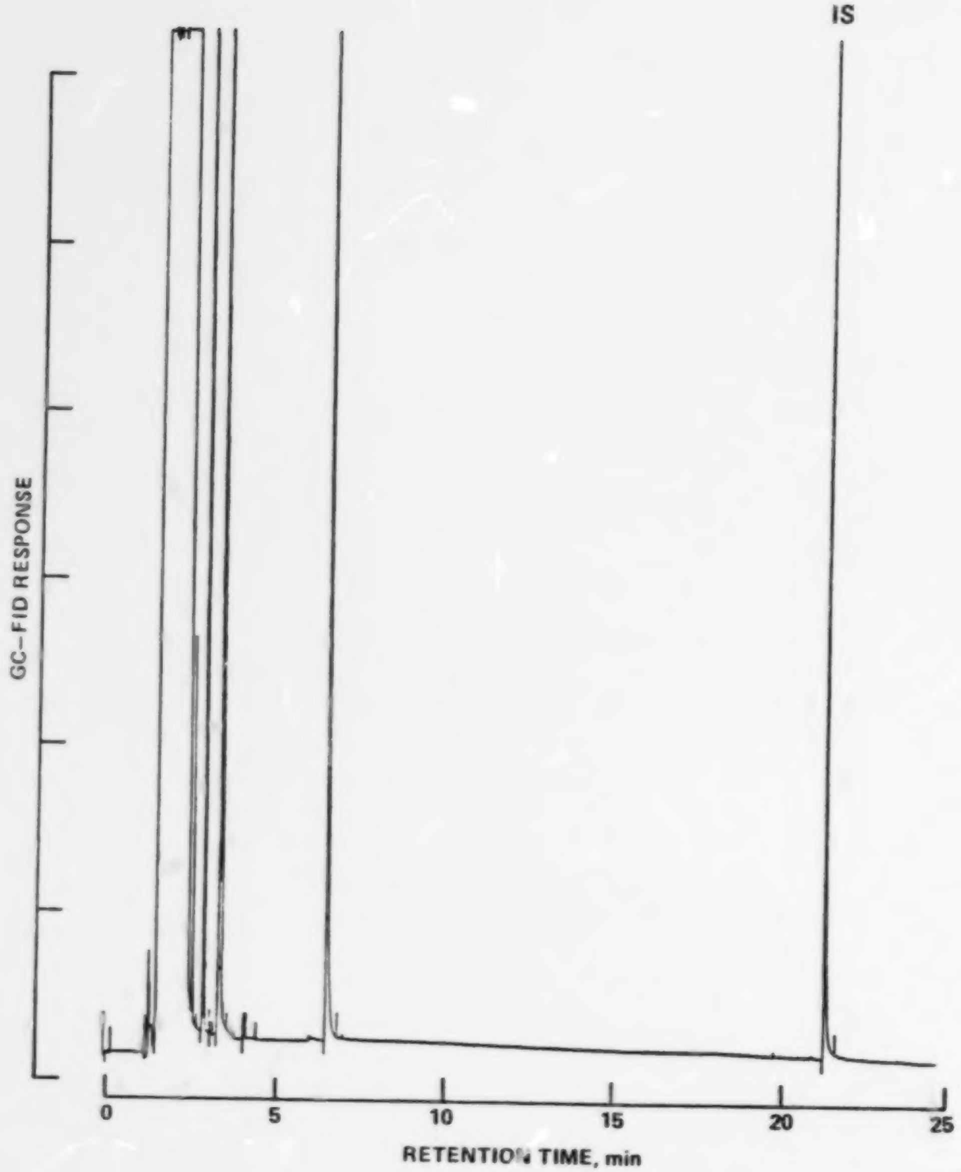

Figure C-12. Chromatogram by GC/FID for chlorobenzene(t_R = 6.9 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

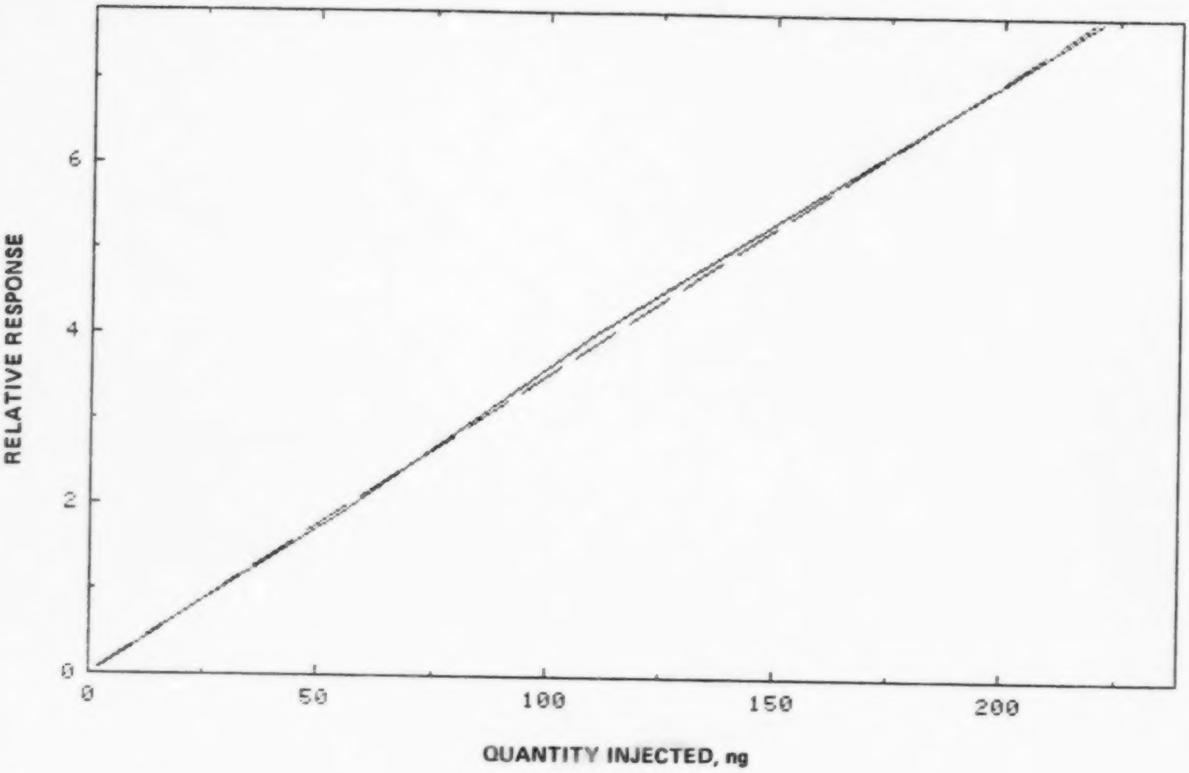

Figure C-13. Calibration curve for the determination of chlorobenzene by GC/FID.

Range: 2 - 220 ng
Correlation coefficient: 0.999
Slope: 3.5×10^{-2}
y-Intercept: 9.3×10^{-3}

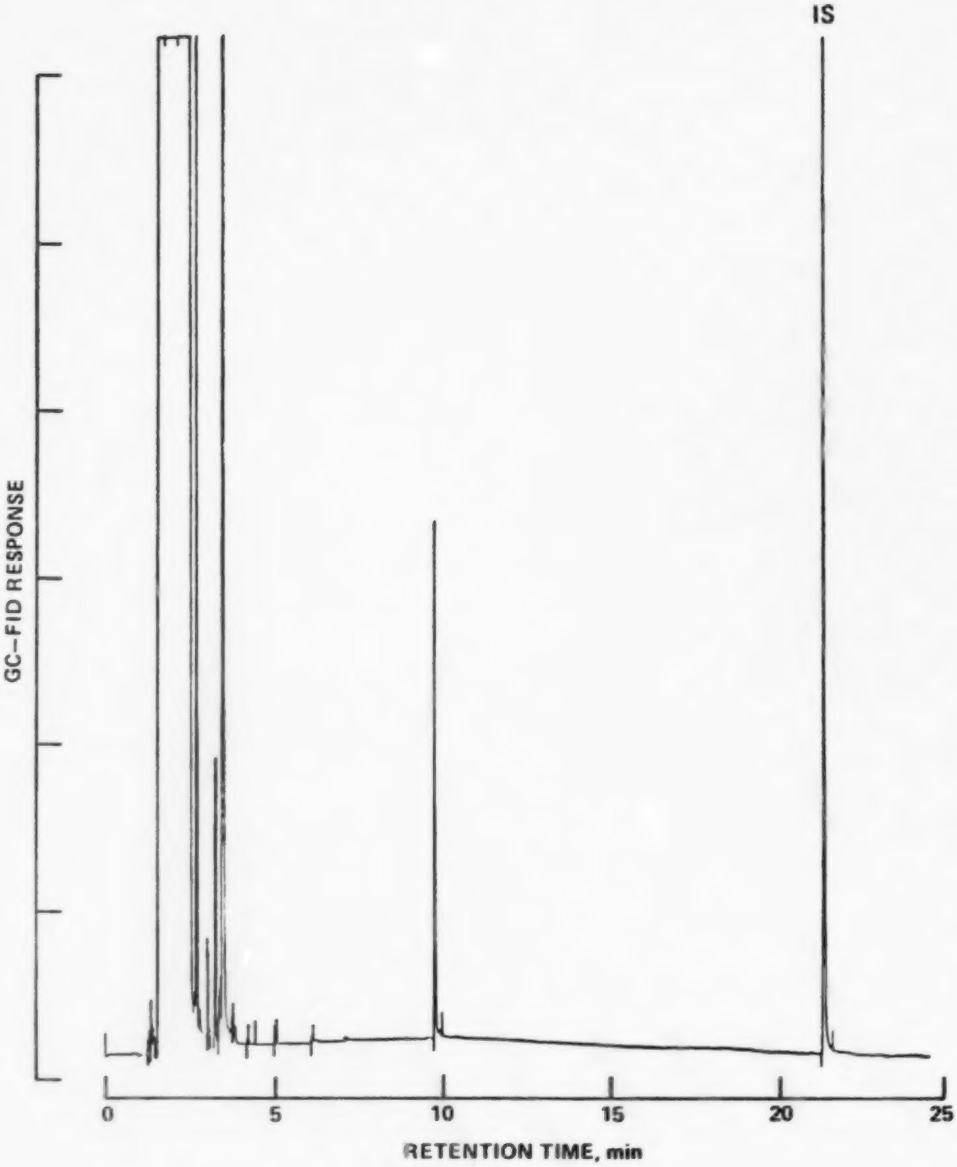

Figure C-14. Chromatogram by GC/FID for m-dichlorobenzene(t_R = 9.9 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

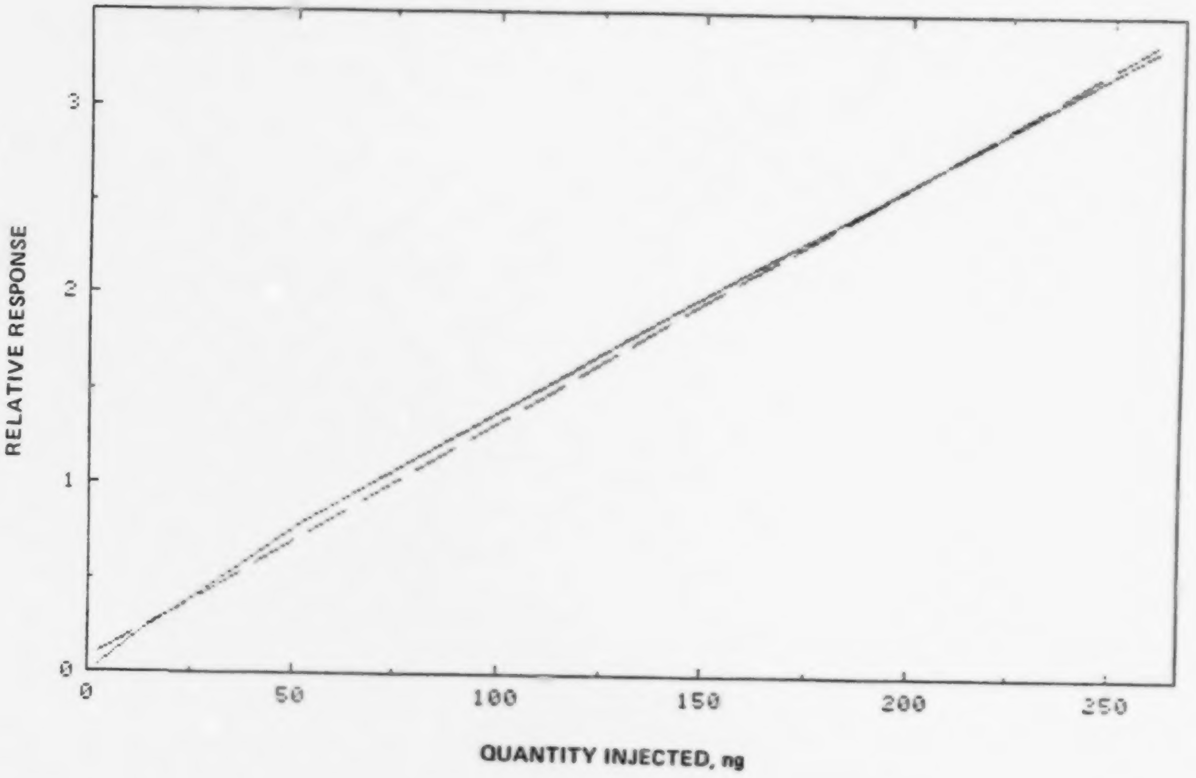

Figure C-15. Calibration curve for the determination of m-dichlorobenzene by GC/FID.

Range: 10 - 260 ng
Correlation coefficient: 0.999
Slope: 4.7×10^{-2}
y-Intercept: -7.8×10^{-2}

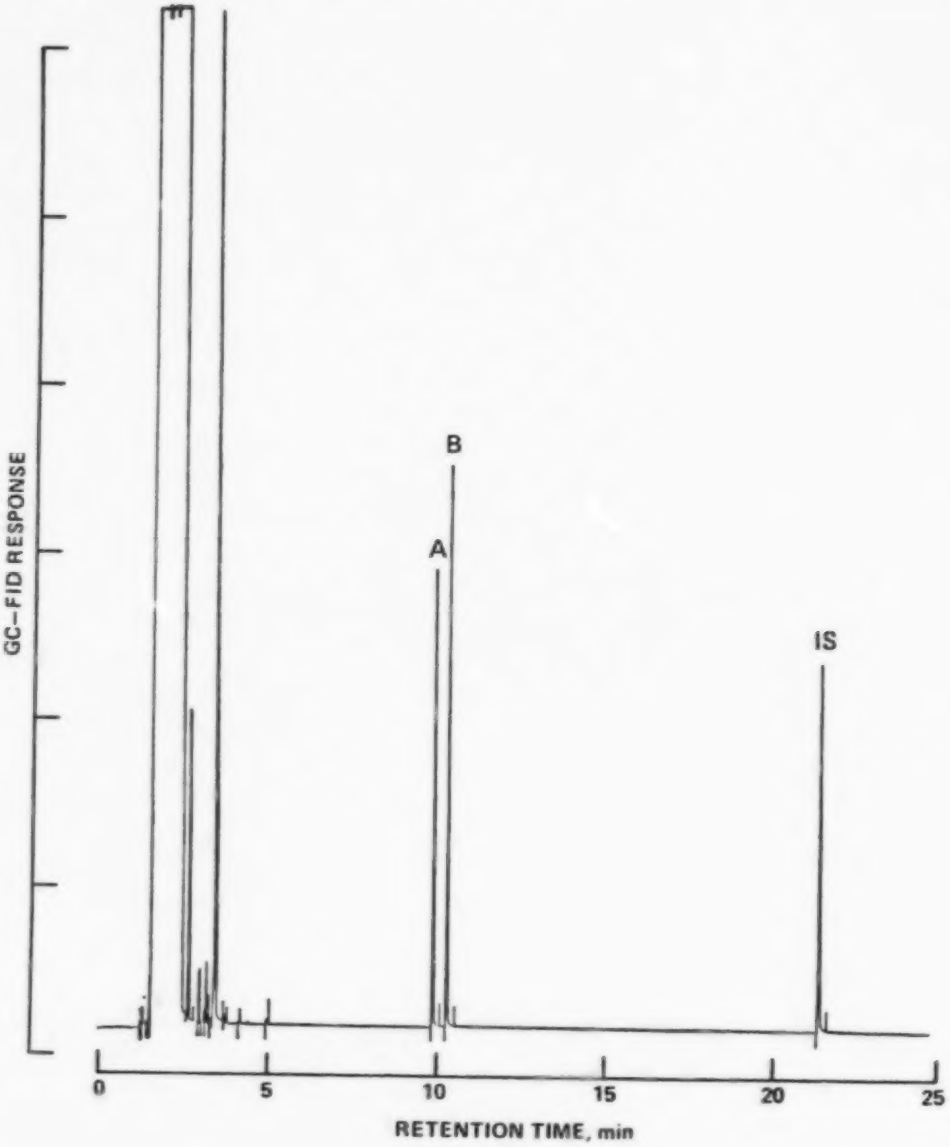

Figure C-16. Chromatogram by GC/FID for

(A) *p*-dichlorobenzene(t_R = 10.0 min) and
(B) *o*-dichlorobenzene(t_R = 10.4 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

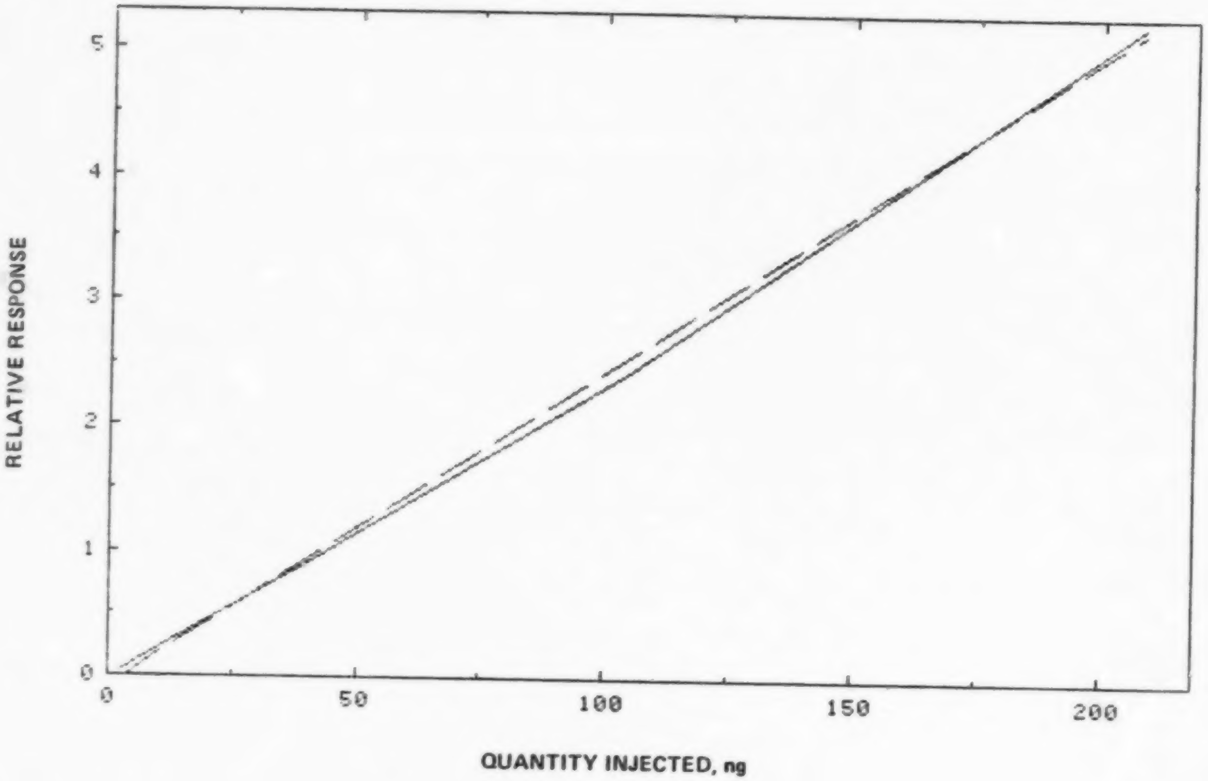

Figure C-17. Calibration curve for the determination of p-dichlorobenzene by GC/FID.

Range: 10 - 210 ng
Correlation coefficient: 0.999
Slope: 5.0×10^{-2}
y-Intercept: -6.7×10^{-2}

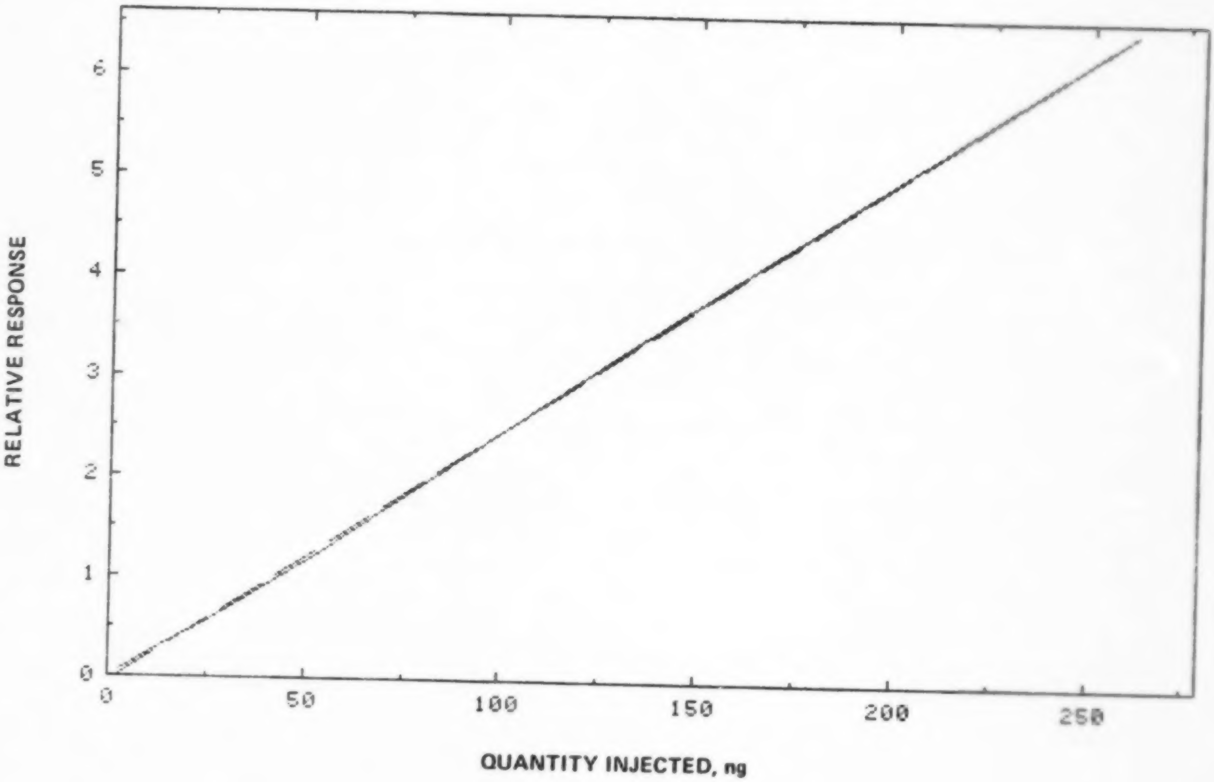

Figure C-18. Calibration curve for the determination of o-dichlorobenzene by GC/FID.

Range: 10 - 260 ng
Correlation coefficient: 0.999
Slope: 2.5×10^{-2}
y-Intercept: -4.3×10^{-2}

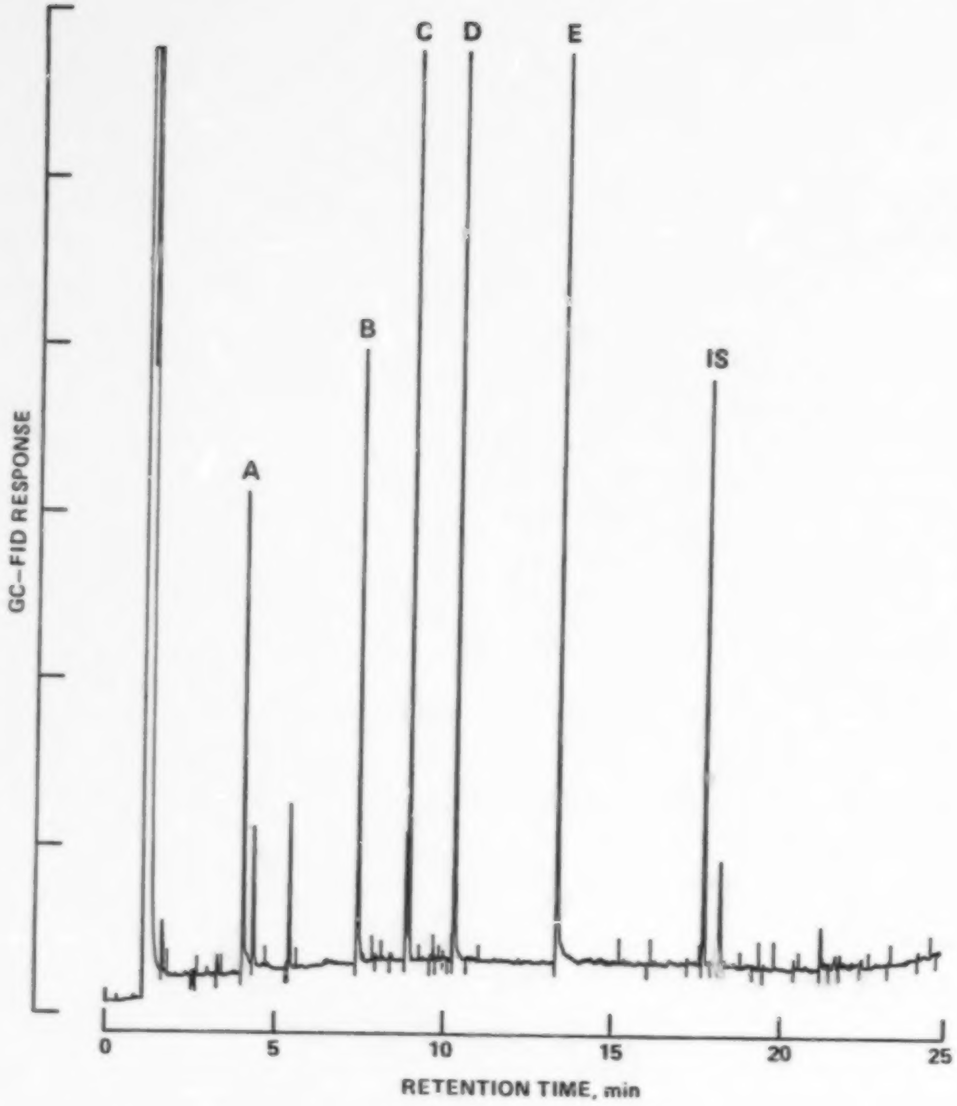

Figure C-19. Chromatogram by GC/FID for

(A) hydroxylamine, N-nitroso-N-phenyl-, ammonium salt(t_R = 4.1 min),
(B) pyridine, 3-chloromethyl-, hydrochloride (t_R = 7.5 min),
(C) o-anisidine hydrochloride(t_R = 9.0 min),
(D) hydroquinone(t_R = 10.4 min), and
(E) o-phenylenediamine, 4-chloro- (t_R = 13.5 min).

Temperature program:
80 °C, 3 min isothermal
80 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

1 to 10 split ratio

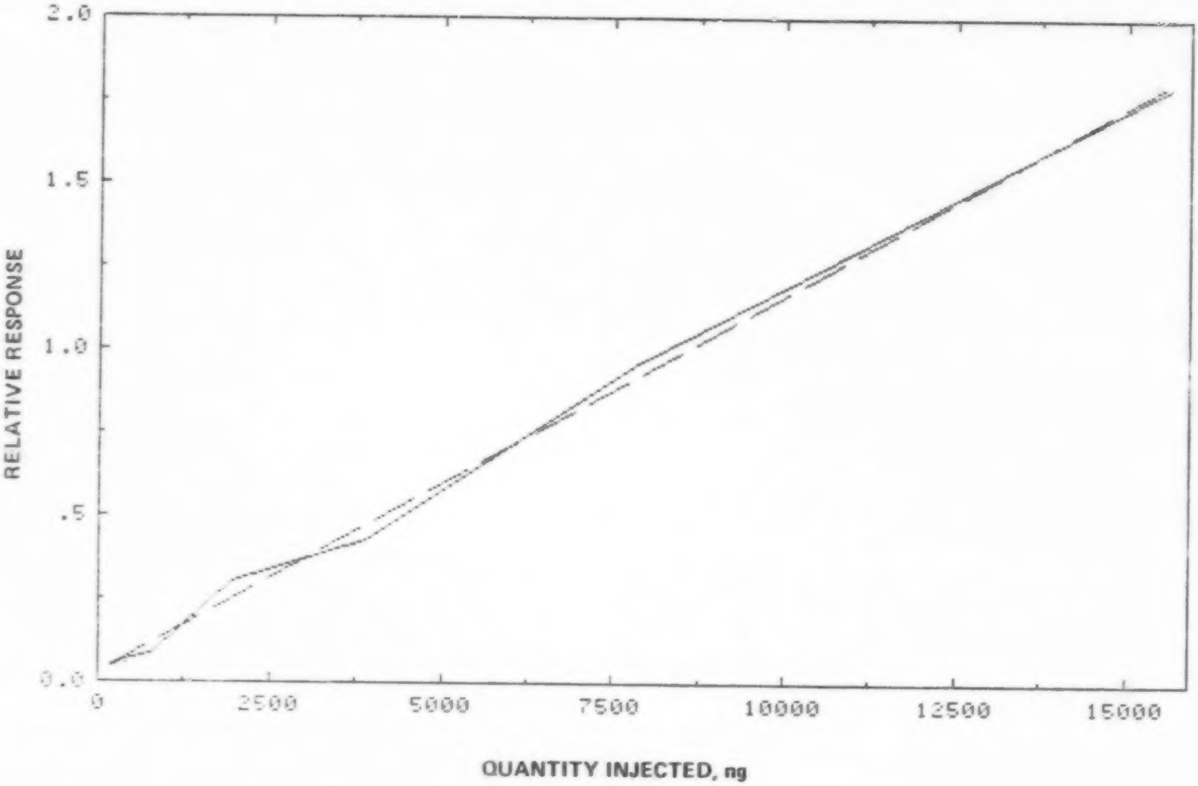

Figure C-20. Calibration curve for the determination of hydroxylamine, N-nitroso-N-phenyl-, ammonium salt by GC/FID.

Range: 200 - 16000 ng
Correlation coefficient: 0.998
Slope: 1.1×10^{-4}
y-Intercept: 2.6×10^{-2}

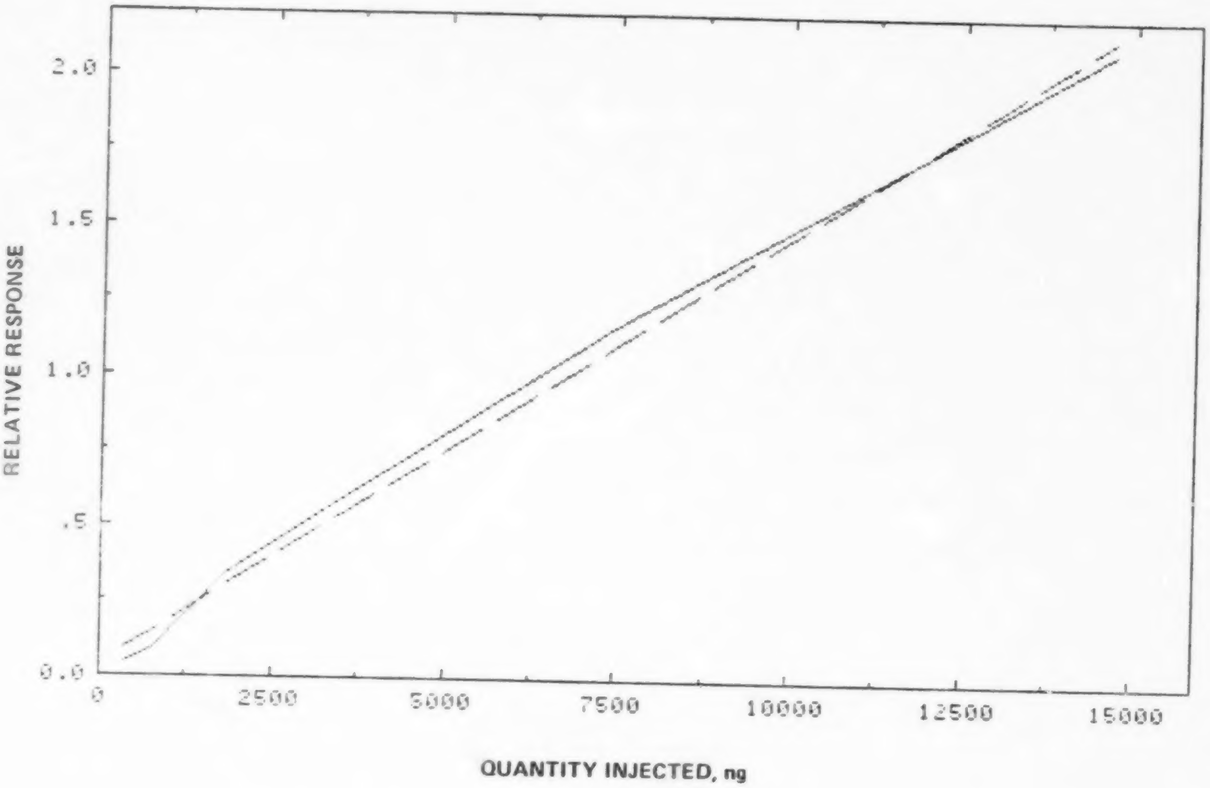

Figure C-21. Calibration curve for the determination of pyridine, 3-chloromethyl-, hydrochloride by GC/FID.

Range: 800 - 15000 ng
Correlation coefficient: 0.997
Slope: 1.0 x 10^{-4}
y-Intercept: 4.0 x 10^{-2}

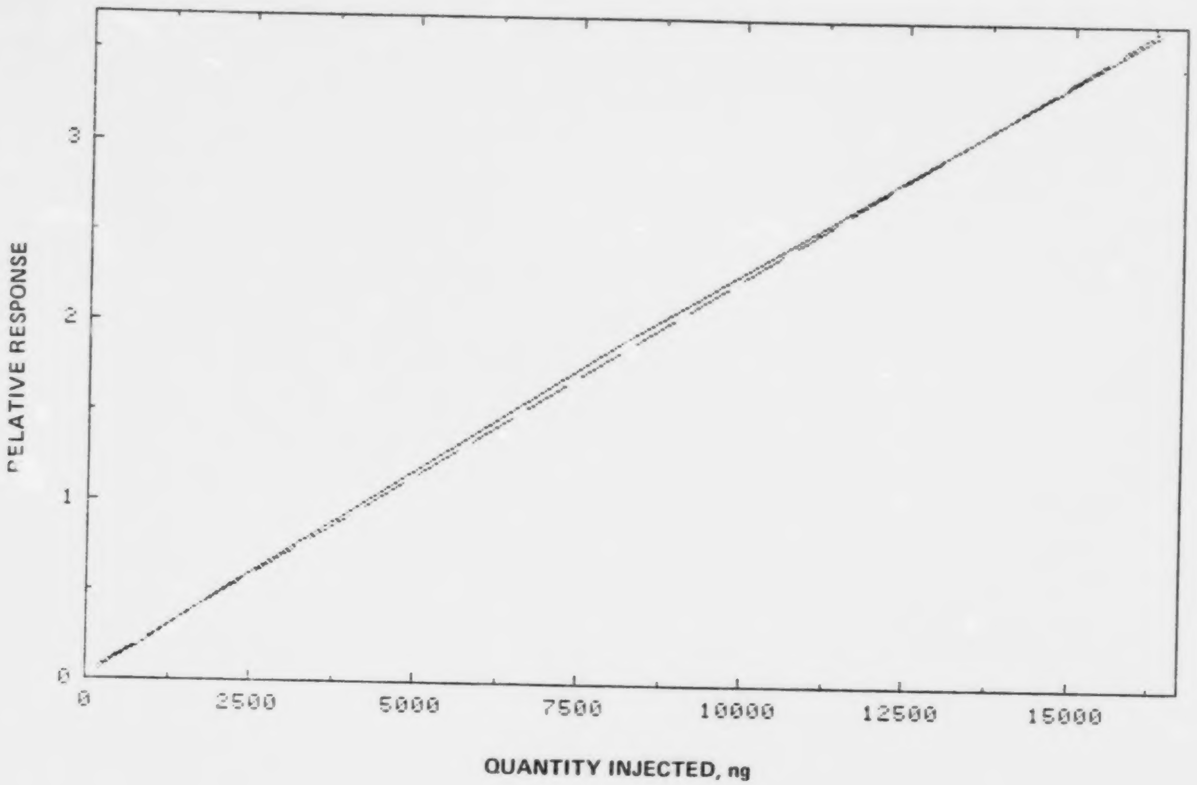

Figure C-22. Calibration curve for the determination of o-anisidine hydrochloride by GC/FID.

Range: 200 - 16000 ng
Correlation coefficient: 0.999
Slope: 2.0×10^{-4}
y-Intercept: 2.7×10^{-2}

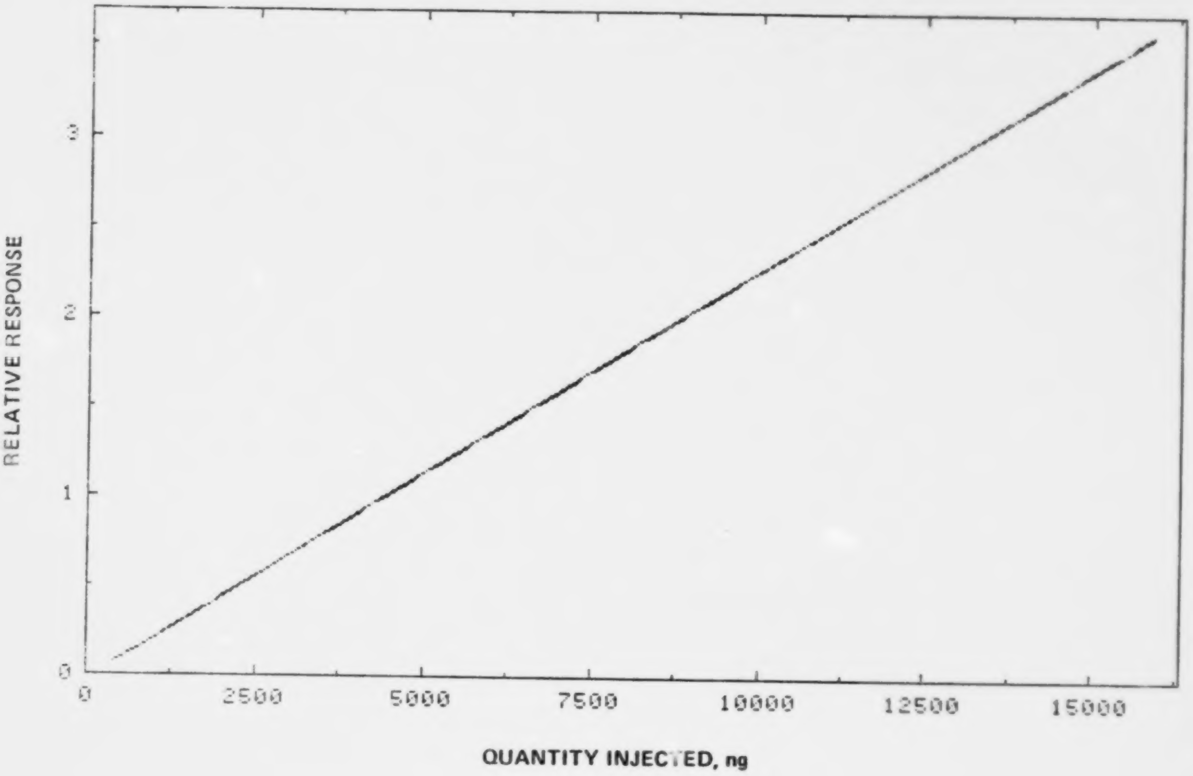

Figure C-23. Calibration curve for the determination of hydroquinone by GC/FID.

Range: 400 - 16000 ng
Correlation coefficient: 0.999
Slope: 2.0×10^{-4}
y-Intercept: -1.7×10^{-4}

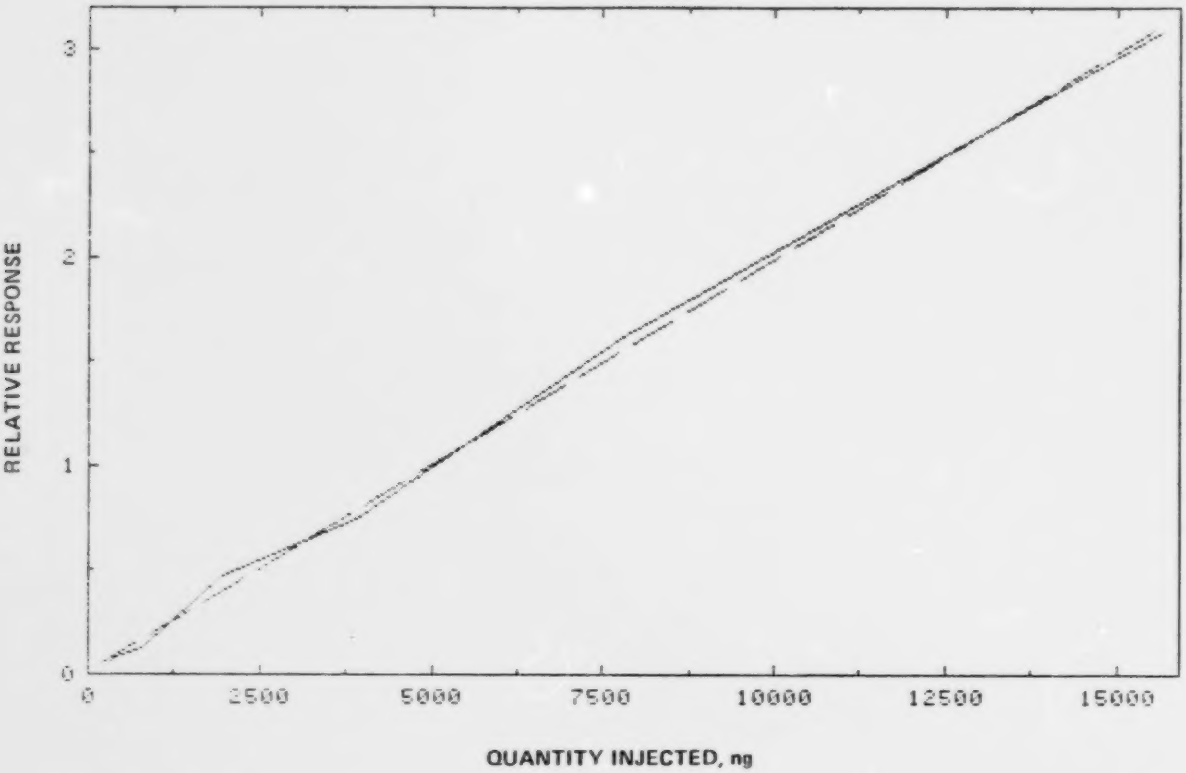

Figure C-24. Calibration curve for the determination of o-phenylenediamine, 4-chloro- by GC/FID.

Range: 200 - 16000 ng
Correlation coefficient: 0.999
Slope: 2.0×10^{-4}
y-Intercept: 1.3×10^{-2}

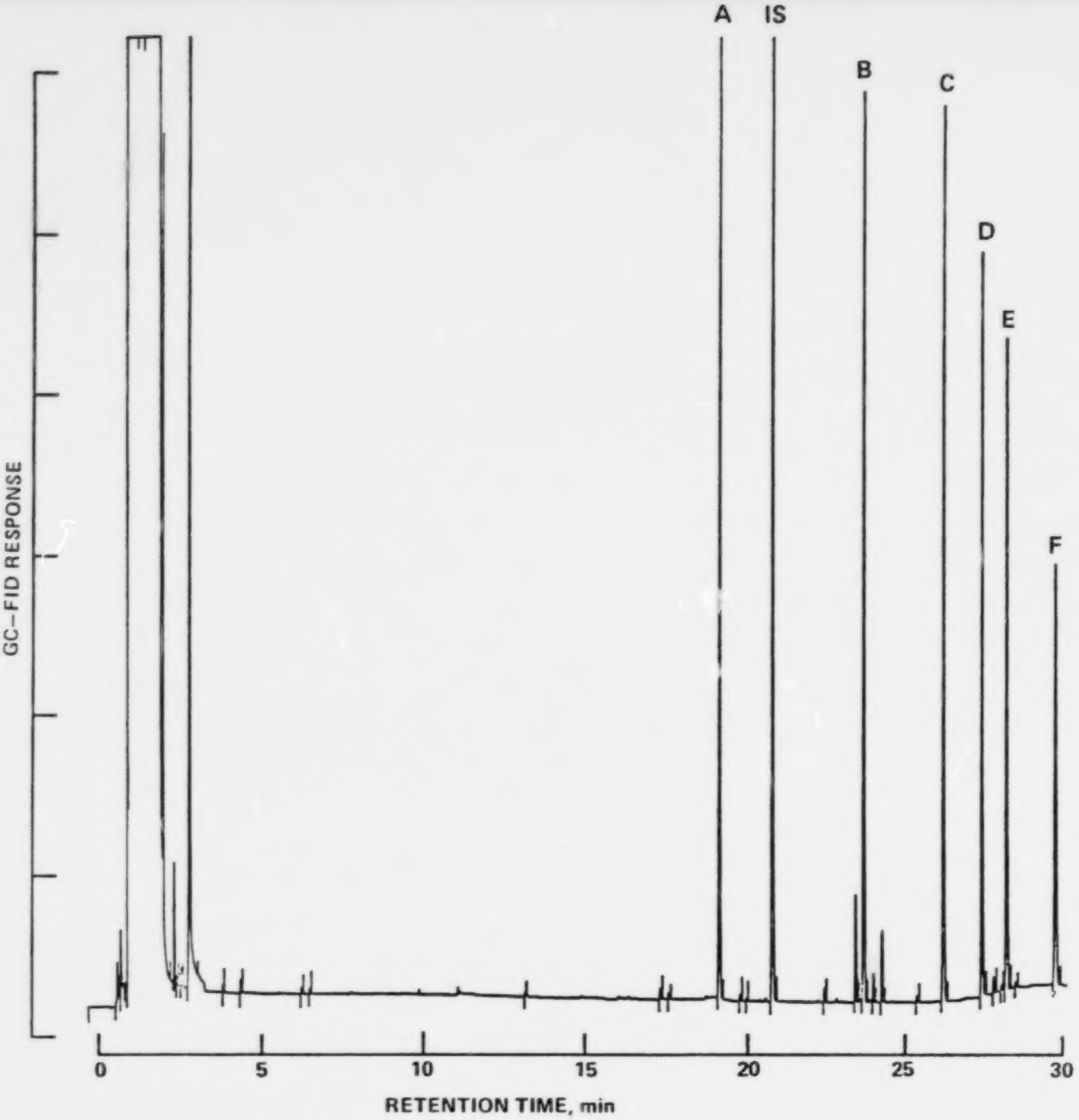

Figure C-25. Chromatogram by GC/FID for

(A) phosphoric acid, dimethyl ester, ester with (E)-3-hydroxy-N,N-dimethylcrotonamide(t_R = 19.0 min),
(B) phosphoric acid, 2-chloro-1-(2,4-dichlorophenyl) vinyl diethyl ester(t_R = 24.0 min),
(C) phosphorodithioic acid, S-(((p-chlorophenyl)thio)methyl) O,O-diethyl ester(t_R = 26.4 min),
(D) phosphorodithioic acid, O,O-dimethyl ester, S-ester with N-(mercaptomethyl)phthalimide(t_R = 27.5 min),
(E) phosphonothioic acid, phenyl-, O-(4-bromo-2,5-dichloro-phenyl) O-methyl ester(t_R = 28.1 min), and
(F) coumarin, 3-chloro-7-hydroxy-4-methyl-, O-ester with O,O-diethyl phosphorothioate(t_R = 30.0 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

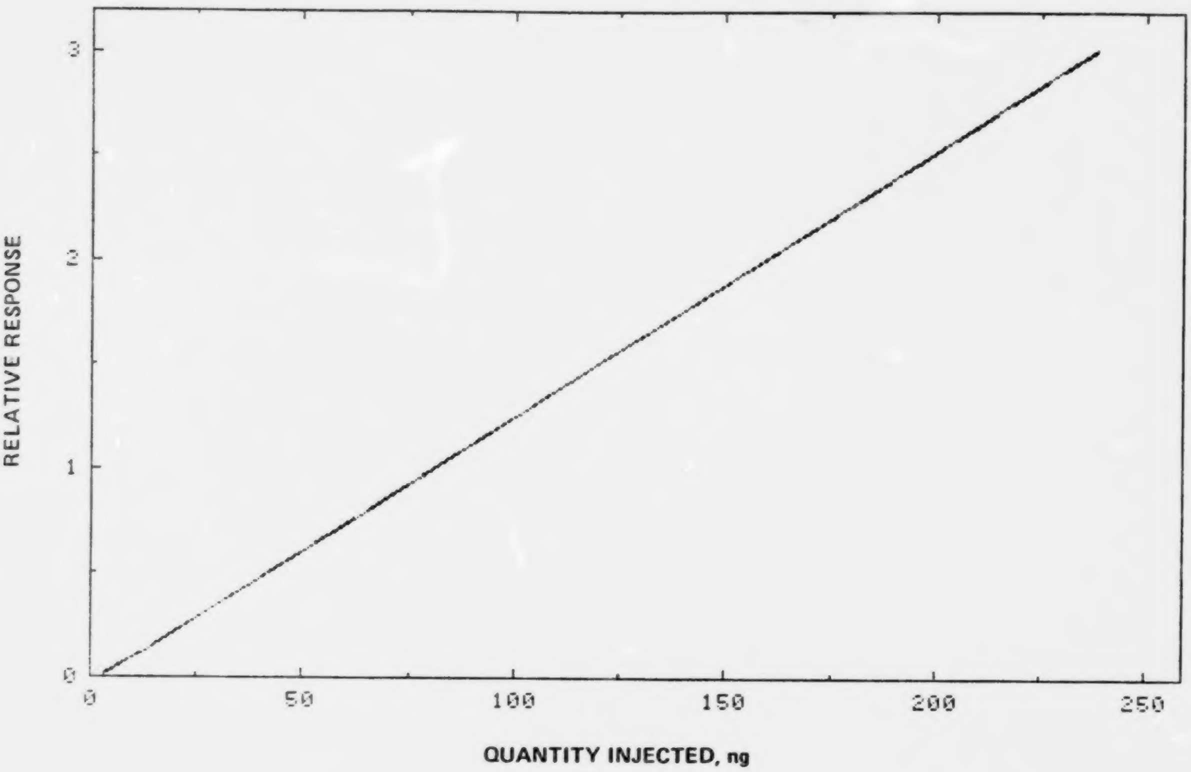

Figure C-26. Calibration curve for the determination of phosphoric acid, dimethyl ester, ester with (E)-3-hydroxy-N,N-dimethylcrotonamide by GC/FID.

Range: 6.0 - 240 ng
Correlation coefficient: 0.999
Slope: 1.3×10^{-2}
y-Intercept: -2.8×10^{-2}

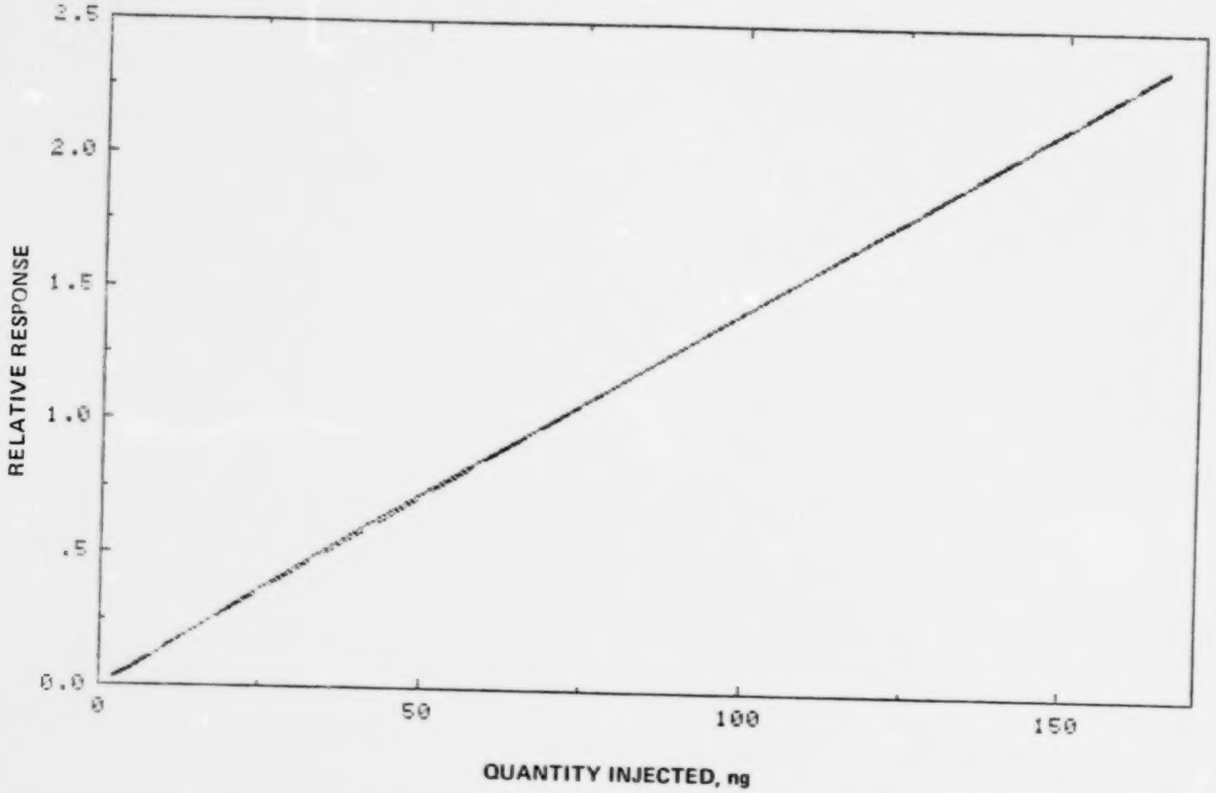

Figure C-27. Calibration curve for the determination of phosphoric acid, 2-chloro-1-(2,4-dichlorophenyl) vinyl diethyl ester by GC/FID.

Range: 2.2 - 170 ng
Correlation coefficient: 0.999
Slope: 1.4×10^{-2}
y-Intercept: 1.5×10^{-3}

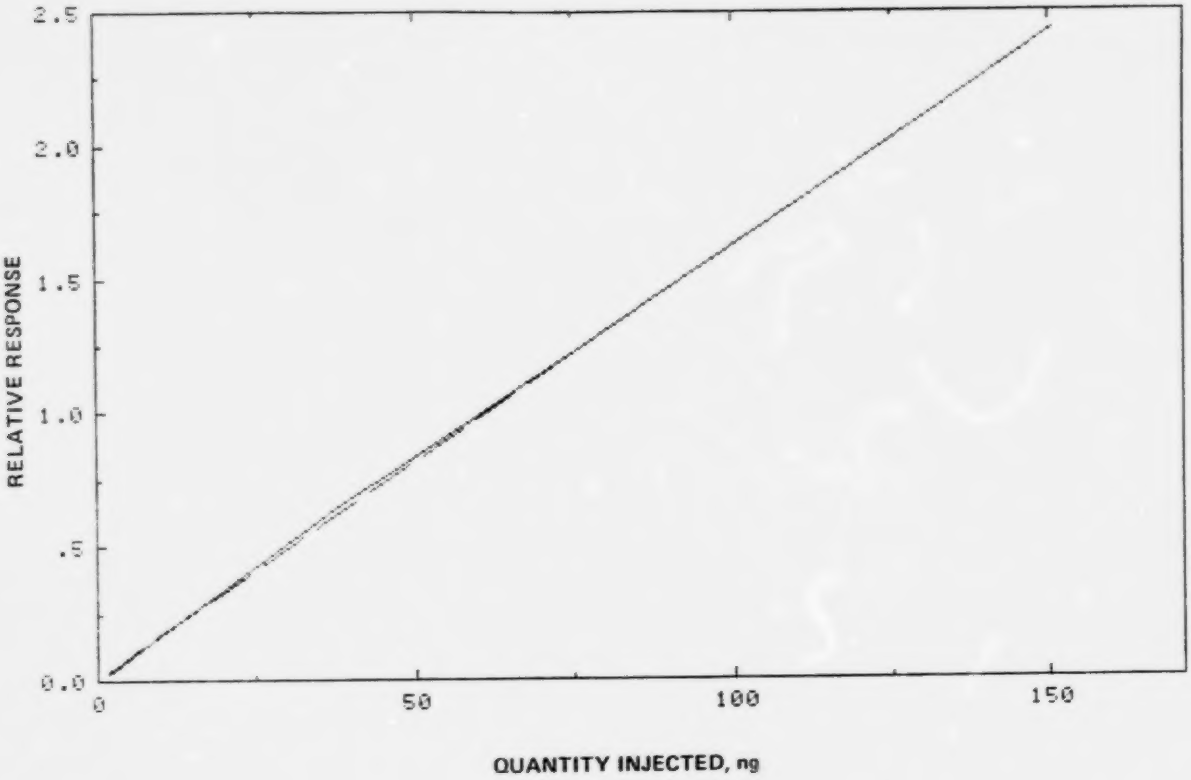

Figure C-28. Calibration curve for the determination of phosphorodithioic acid, S-(((p-chlorophenyl)thio)methyl) O,O-diethyl ester by GC/FID.

Range: 7.5 - 150 ng
Correlation coefficient: 0.999
Slope: 1.5×10^{-2}
y-Intercept: 1.2×10^{-1}

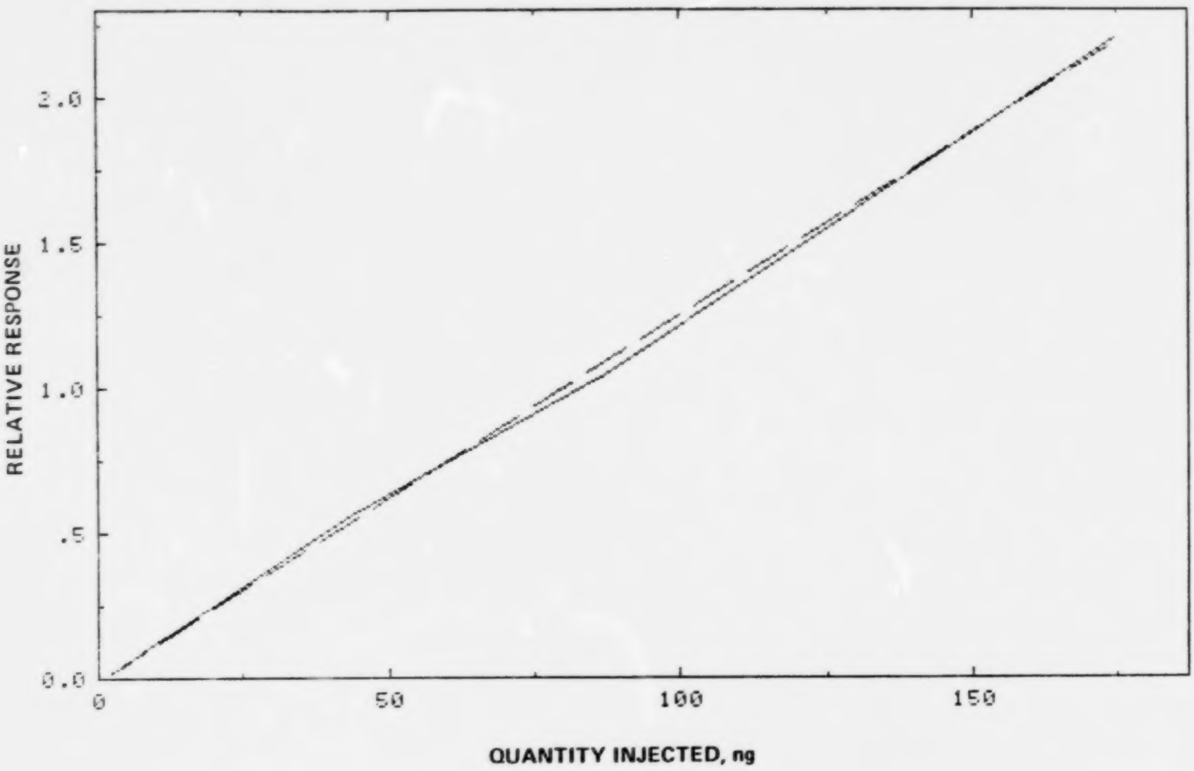

Figure C-29. Calibration curve for the determination of phosphorodithioic acid, O,O-dimethyl ester, S-ester with N-(mercaptomethyl)phthalimide by GC/FID.

Range: 2.2 - 170 ng
Correlation coefficient: 0.999
Slope: 1.3 x 10^{-2}
y-Intercept: -8.7 x 10^{-3}

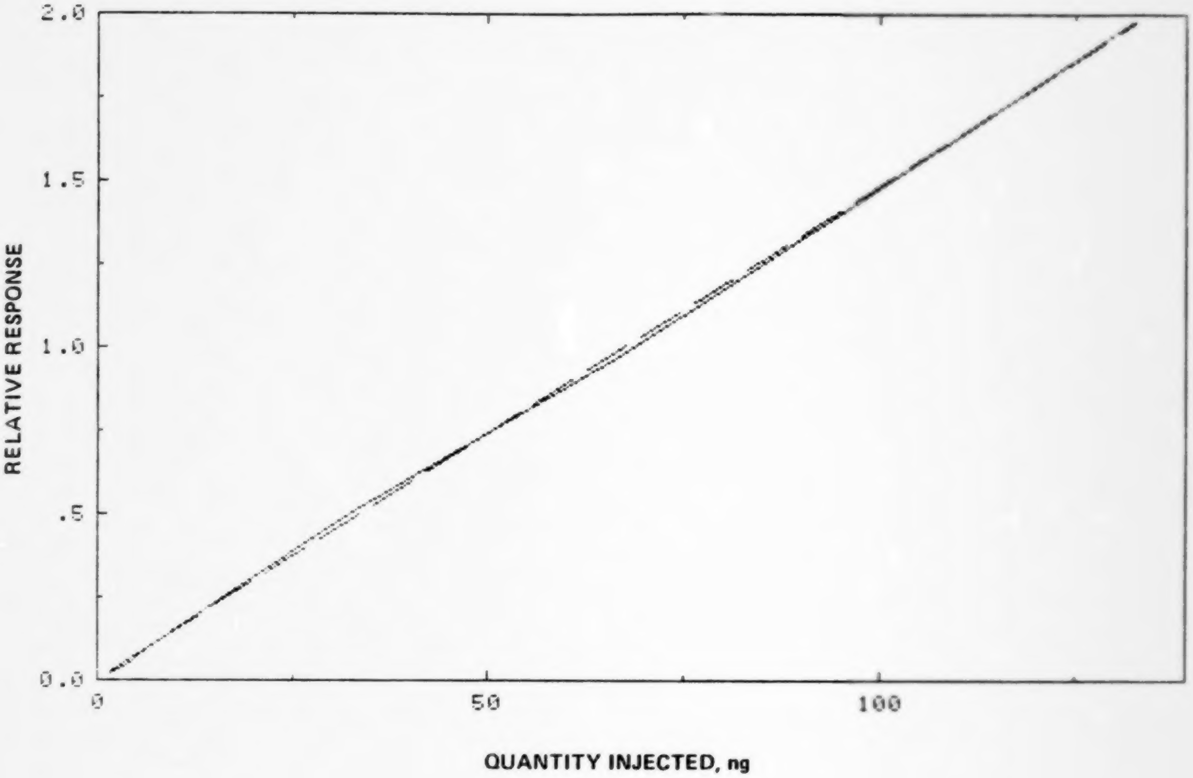

Figure C-30. Calibration curve for the determination of phosphonothioic acid, phenyl-, O-(4-bromo-2,5-dichlorophenyl) O-methyl ester by GC/FID.

Range: 1.7 - 130 ng
Correlation coefficient: 0.999
Slope: 1.5×10^{-2}
y-Intercept: 5.3×10^{-4}

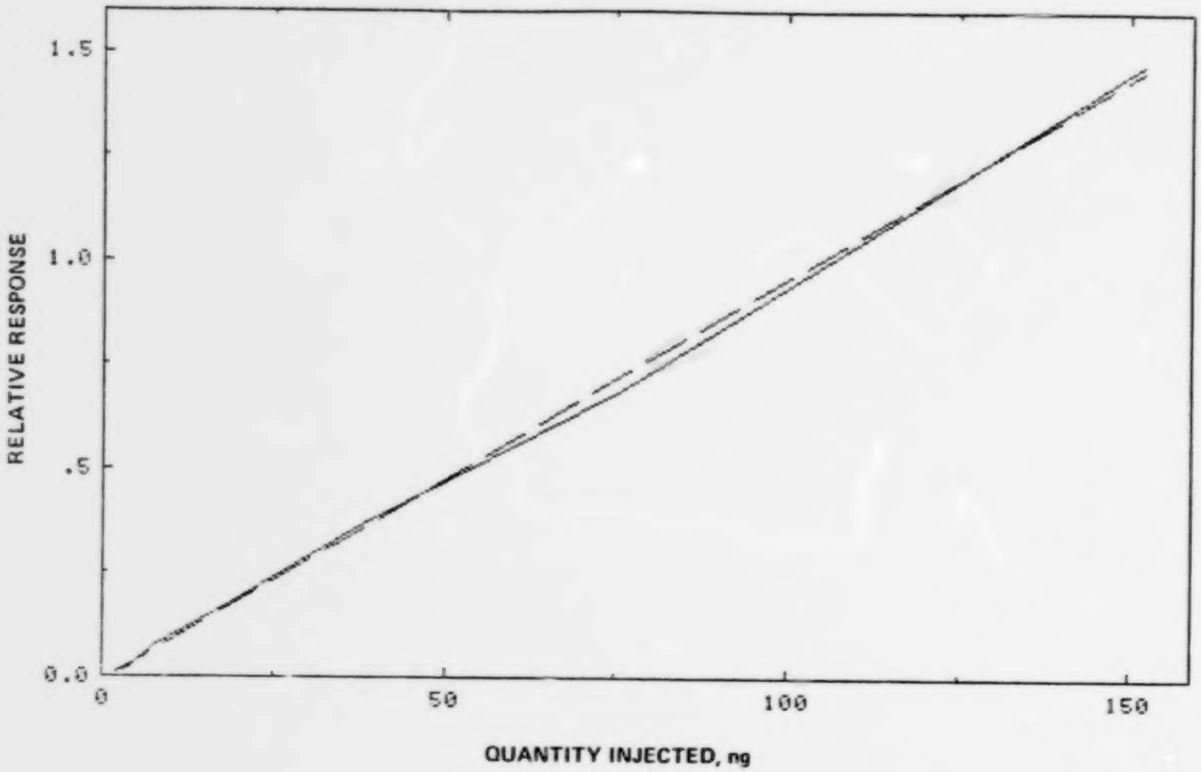

Figure C-31. Calibration curve for the determination of coumarin, 3-chloro-7-hydroxy-4-methyl-, O-ester with O,O-diethyl phosphorothioate by GC/FID.

Range: 1.9 - 150 ng
Correlation coefficient: 0.999
Slope: 9.7×10^{-3}
y-Intercept: -1.0×10^{-2}

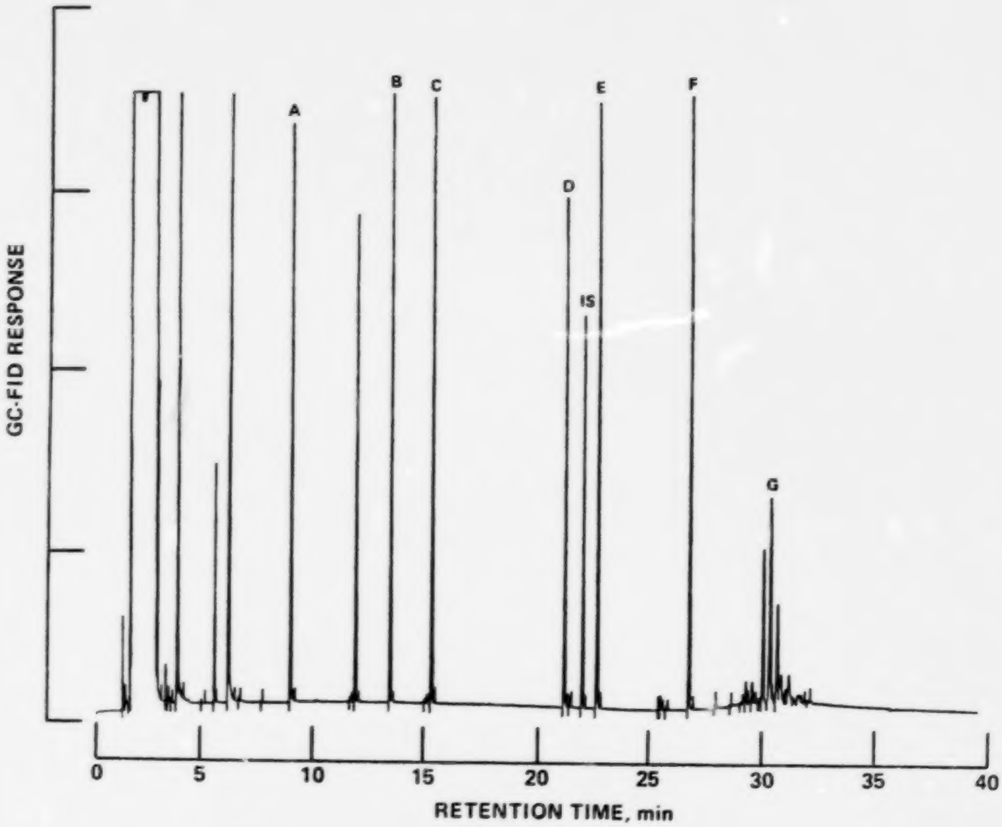

Figure C-32. Chromatogram by GC/FID for

(A) phosphoric acid, trimethyl ester(t_R = 8.8 min),
(B) o-anisidine(t_R = 13.3 min),
(C) aniline, 2,4,5-trimethyl- (t_R = 15.2 min),
(D) o-anisidine, 5-nitro- (t_R = 21.3 min),
(E) biphenyl, 4-nitro(t_R = 22.8 min),
(F) o-toluidine, 4-(o-tolylazo)- (t_R = 27.0 min), and
(G) phosphoric acid, tri-o-tolyl ester(tR = 30.7 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

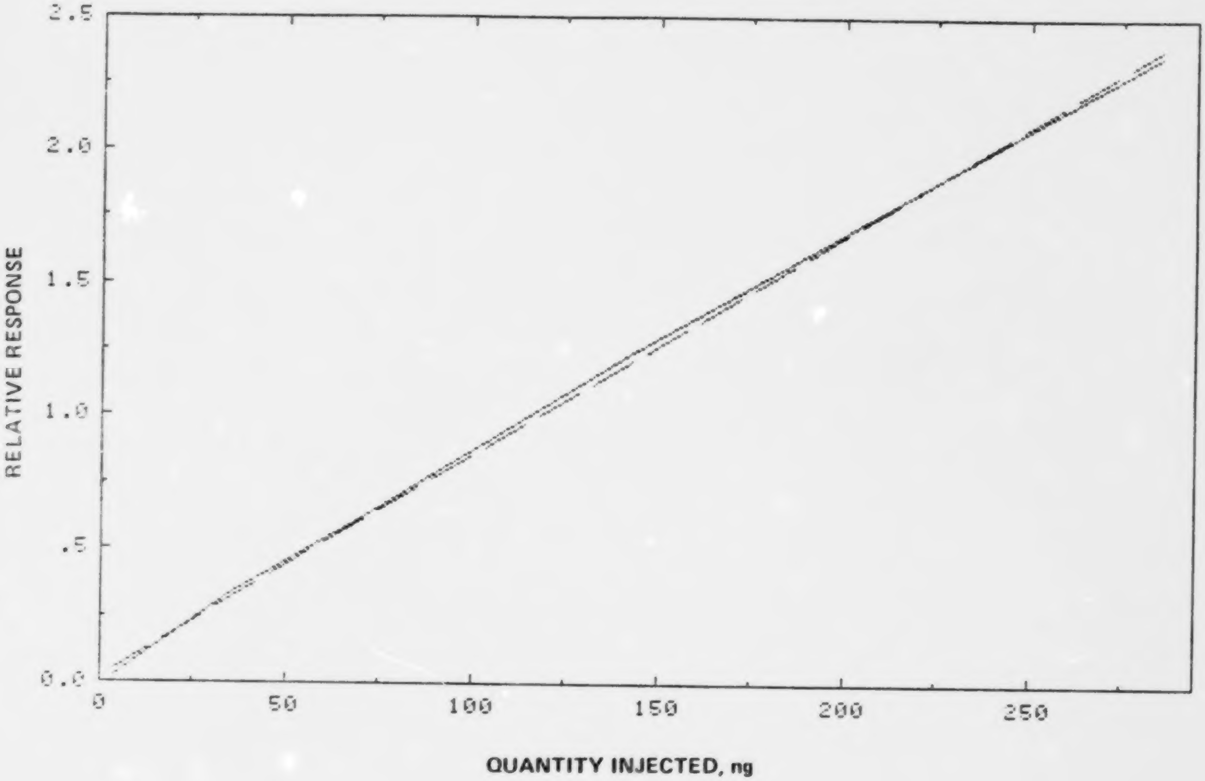

Figure C-33. Calibration curve for the determination of phosphoric acid, trimethyl ester by GC/FID.

Range: 14 - 280 ng
Correlation coefficient: 0.999
Slope: 8.3×10^{-3}
y-Intercept: 2.4×10^{-2}

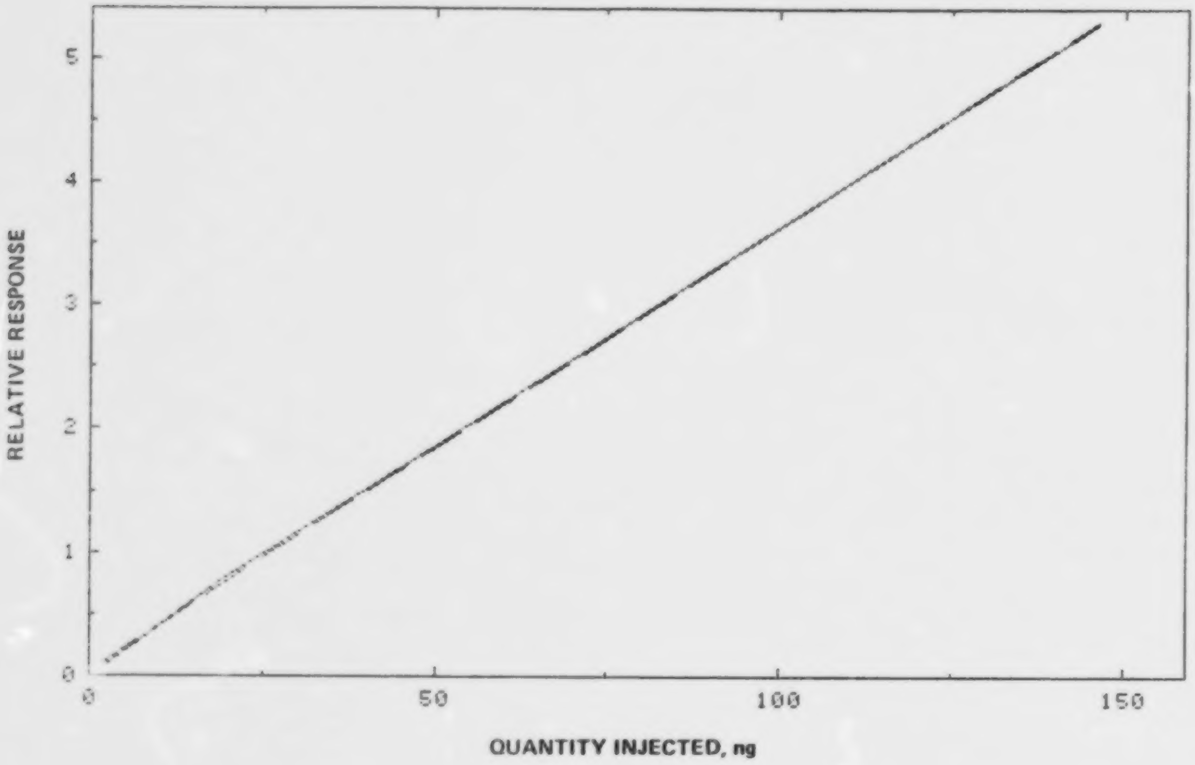

Figure C-34. Calibration curve for the determination of *o*-anisidine by GC/FID.

Range: 7.3 - 150 ng
Correlation coefficient: 0.999
Slope: 3.6×10^{-2}
y-Intercept: 4.8×10^{-2}

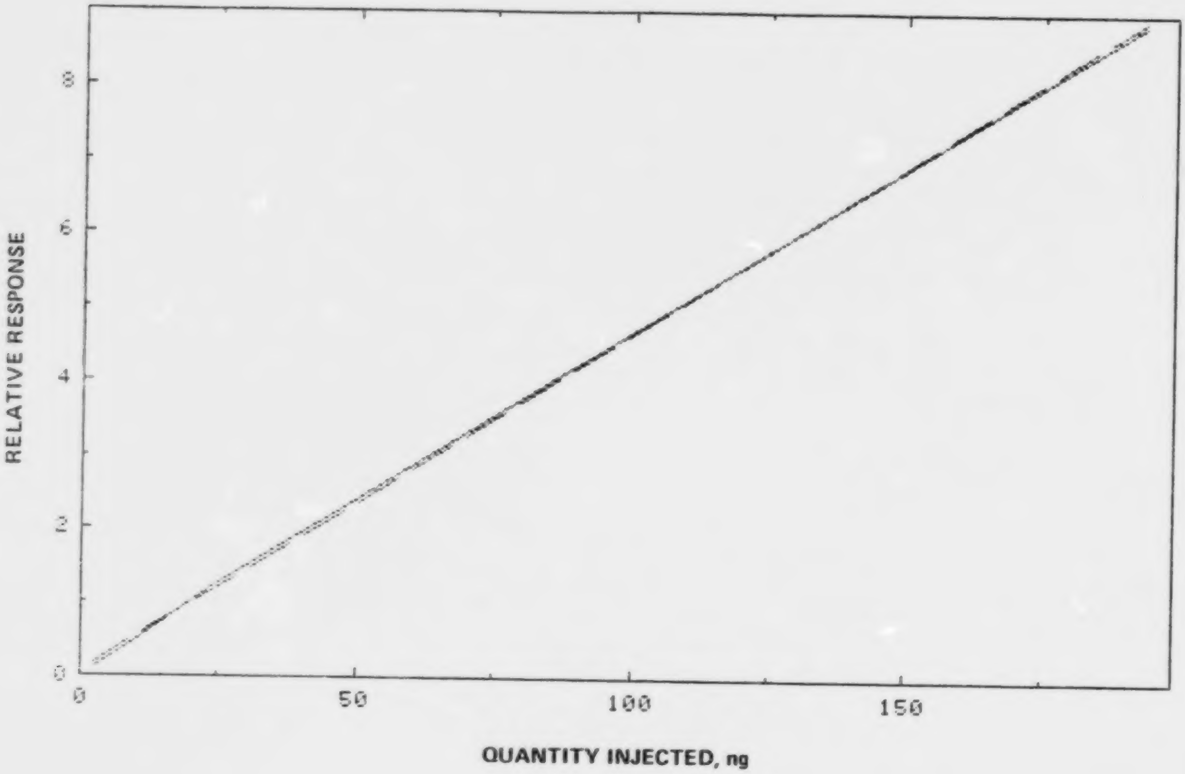

Figure C-35. Calibration curve for the determination of aniline, 2,4,5-trimethyl- by GC/FID.

Range: 9.3 - 190 ng
Correlation coefficient: 0.999
Slope: 4.6×10^{-2}
y-Intercept: 8.2×10^{-2}

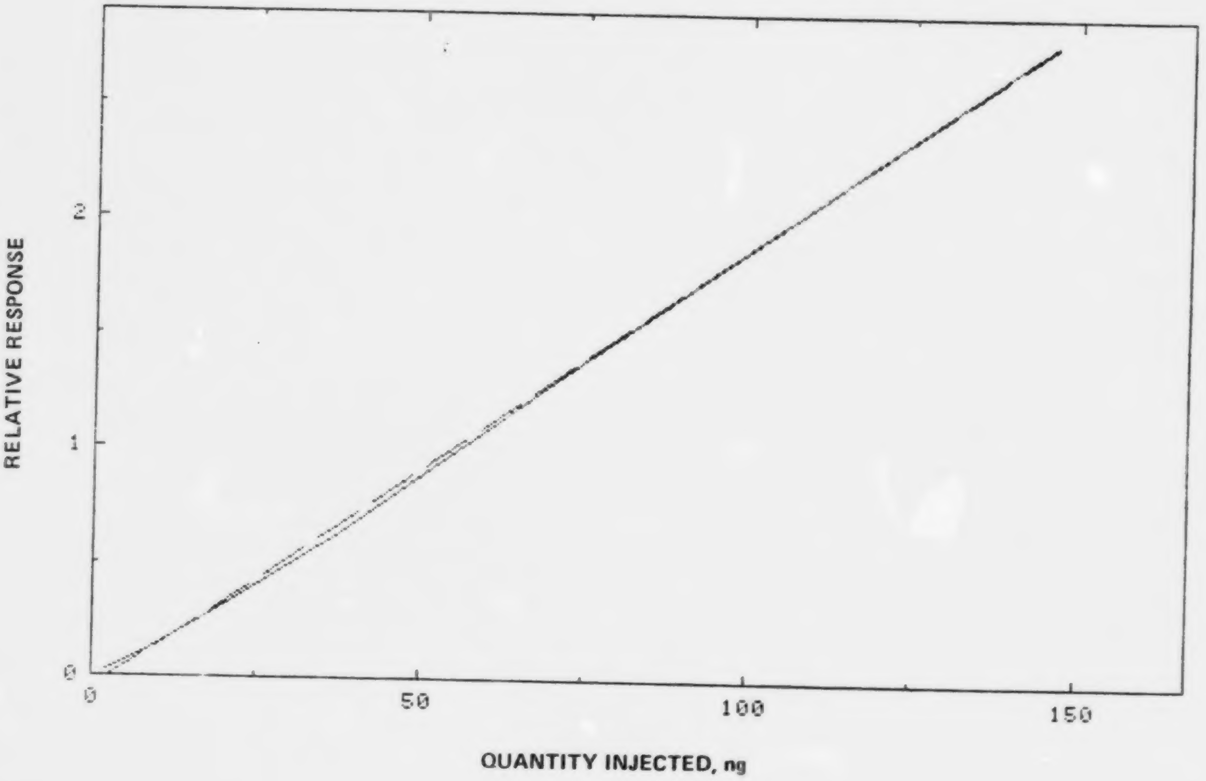

Figure C-36. Calibration curve for the determination of o-anisidine, 5-nitro- by GC/FID.

Range: 7.3 - 150 ng
Correlation coefficient: 0.999
Slope: 1.9×10^{-2}
y-Intercept: -5.3×10^{-2}

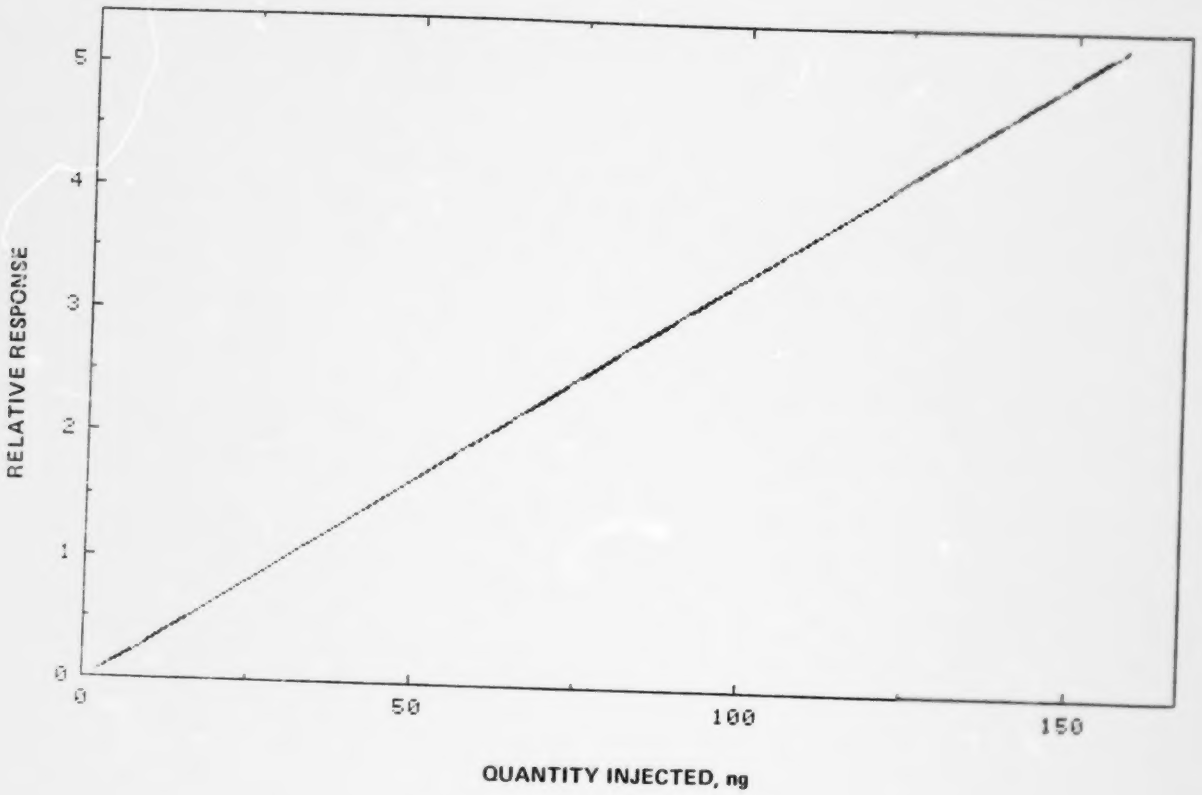

Figure C-37. Calibration curve for the determination of biphenyl, 4-nitro by GC/FID.

Range: 7.9 - 160 ng
Correlation coefficient: 0.999
Slope: 3.4×10^{-2}
y-Intercept: -1.7×10^{-2}

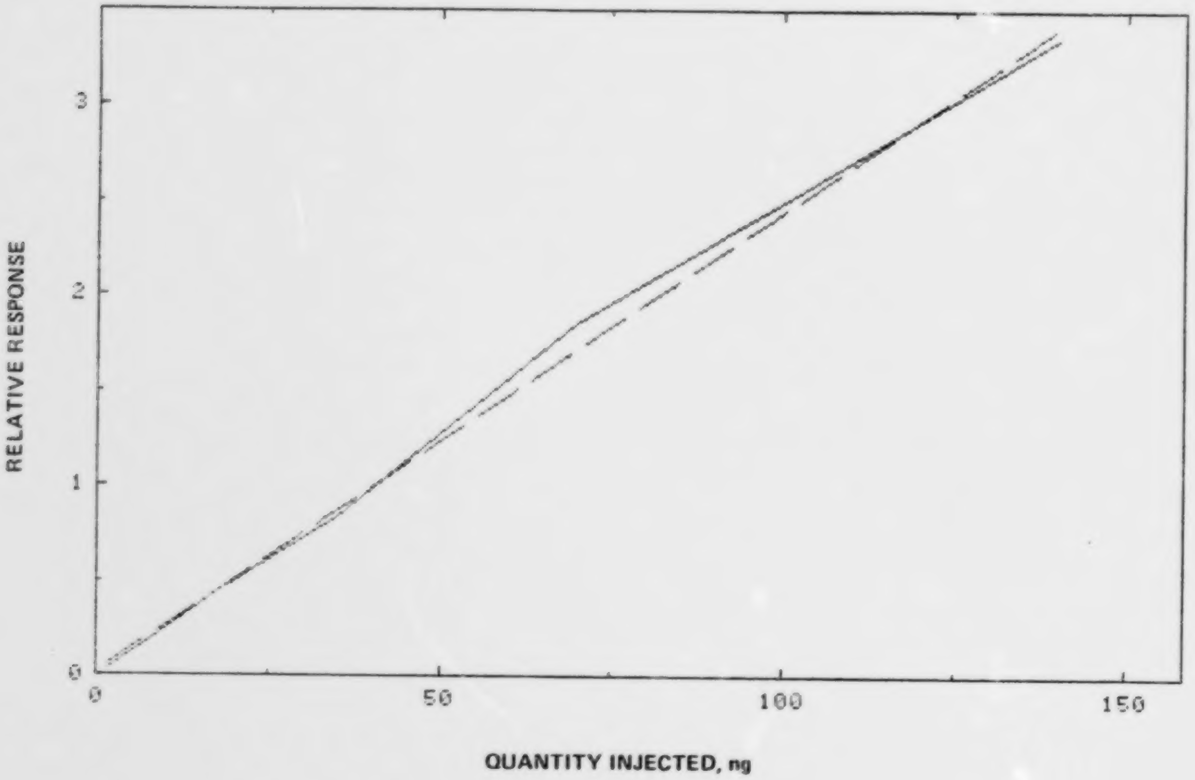

Figure C-38. Calibration curve for the determination of o-toluidine, 4-(o-tolylazo)- by GC/FID.

Range: 7.0 - 140 ng
Correlation coefficient: 0.998
Slope: 2.4×10^{-2}
y-Intercept: 1.2×10^{-2}

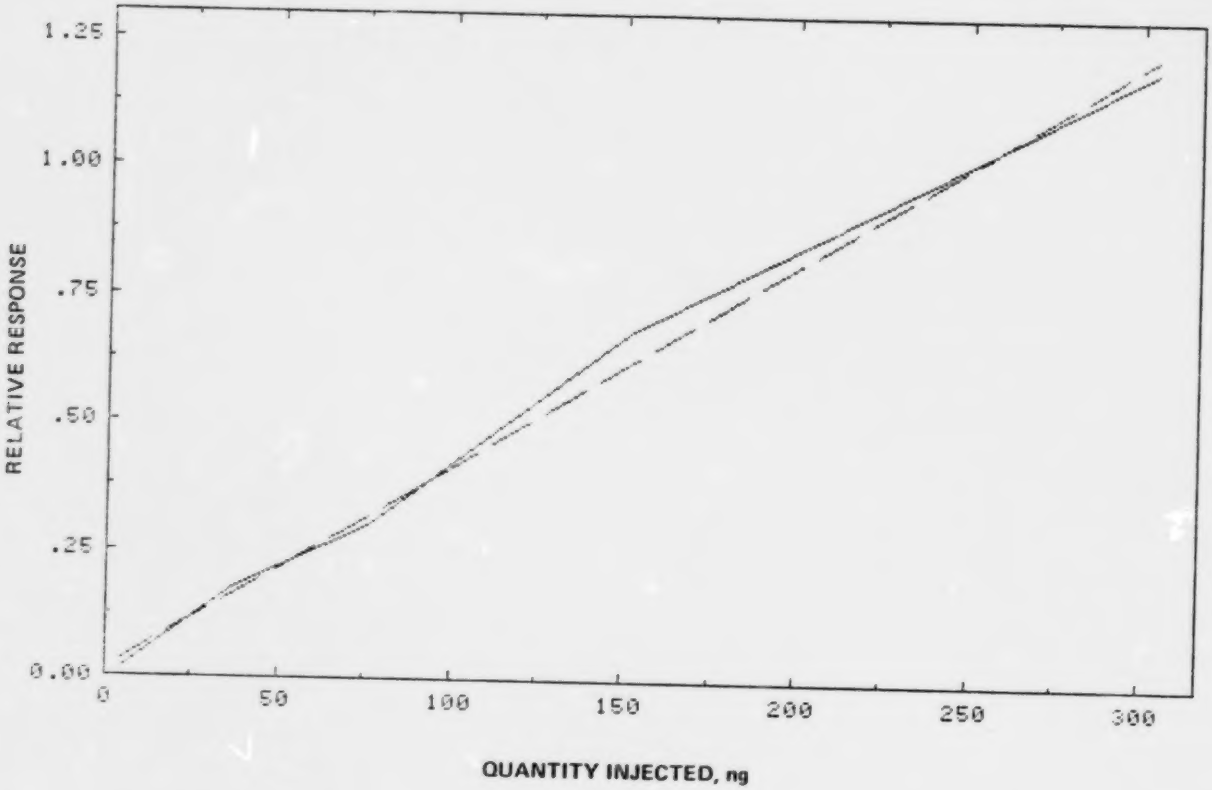

Figure C-39. Calibration curve for the determination of phosphoric acid, tri-o-tolyl ester by GC/FID.

Range: 15 - 300 ng
Correlation coefficient: 0.998
Slope: 4.0×10^{-3}
y-Intercept: 1.5×10^{-2}

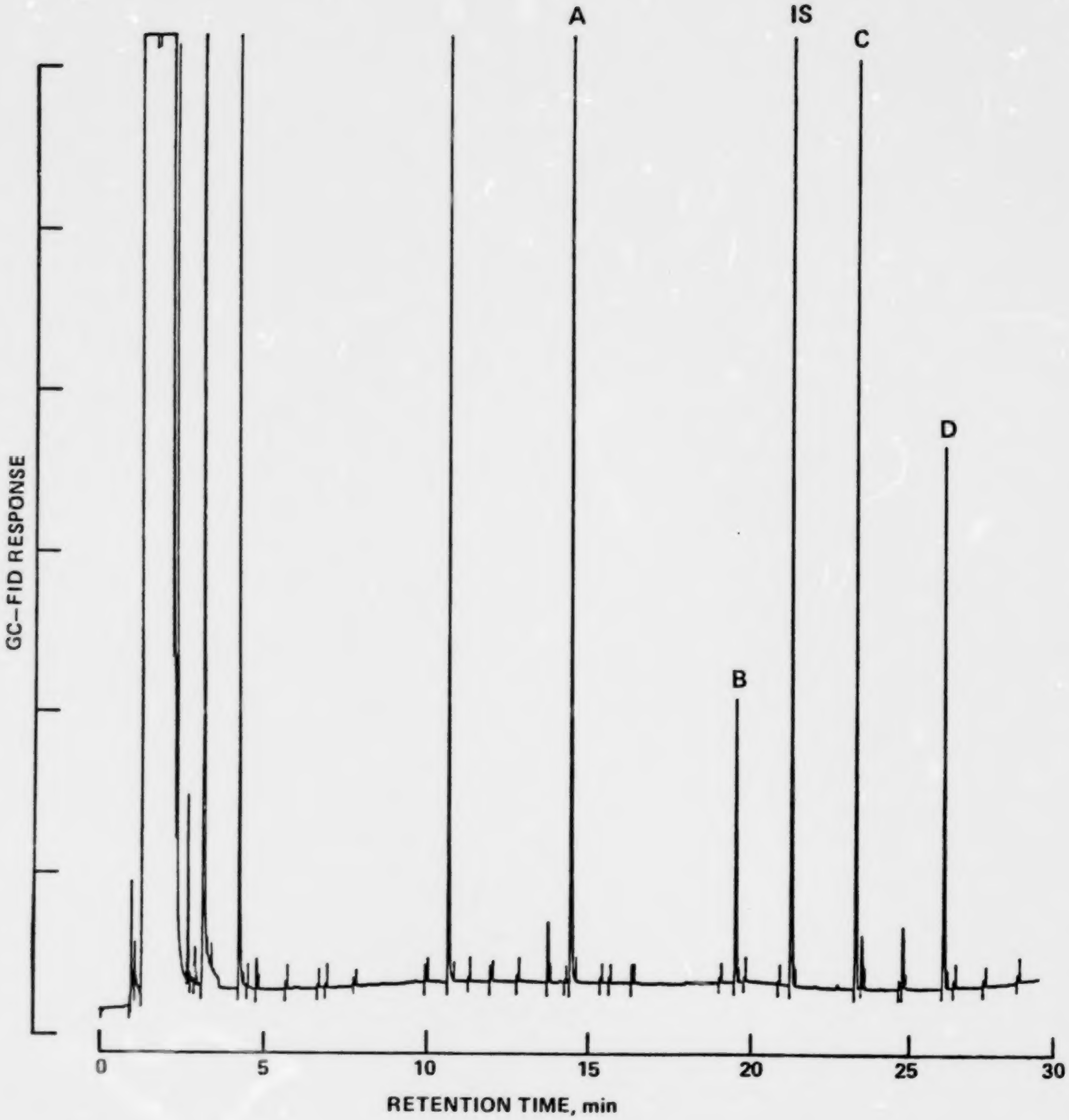

Figure C-40. Chromatogram by GC/FID for

(A) phosphoric triamide, hexamethyl- (t_R = 14.9 min)
(B) phosphoric acid, 1,2-dibromo-2,2-dichloroethyl dimethyl ester(t_R = 19.9 min),
(C) phosphorothioic acid, O,O-dimethyl-, O-(4-(methylthio)-m-tolyl) ester(t_R = 23.7 min)
(D) phosphorothioic acid, O,O-diethyl O-(p-(methylsulfinyl)phenyl) ester(t_R = 26.4 min)

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

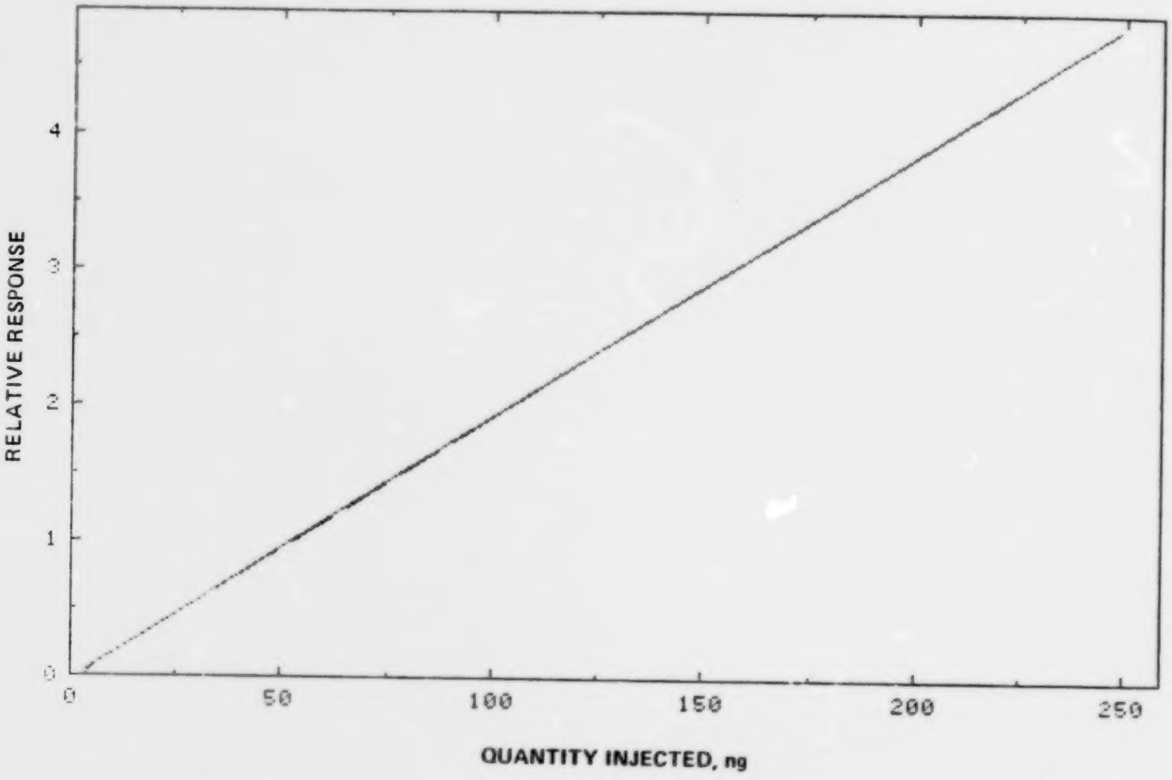

Figure C-41. Calibration curve for the determination of phosphoric triamide, hexamethyl- by GC/FID.

Range: 3.1 - 250 ng
Correlation coefficient: 0.999
Slope: 1.9×10^{-2}
y-Intercept: -2.3×10^{-2}

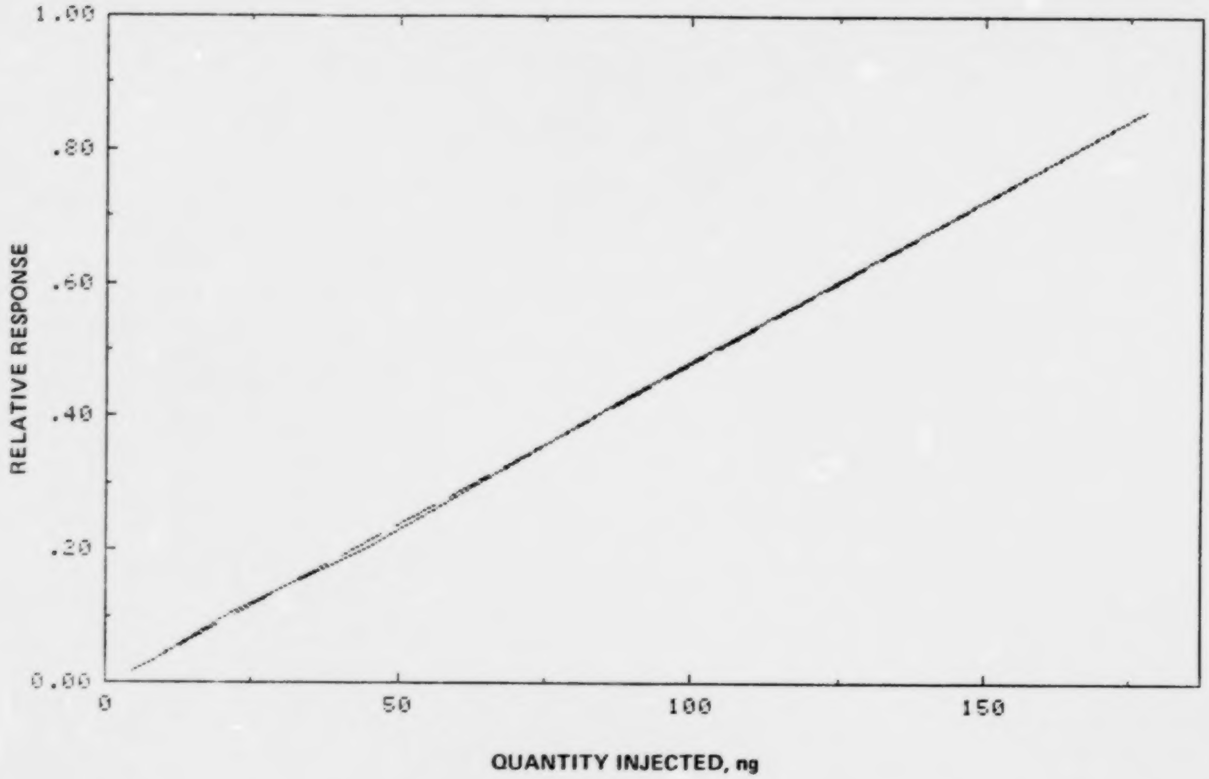

Figure C-42. Calibration curve for the determination of phosphoric acid, 1,2-dibromo-2,2-dichloroethyl dimethyl ester by GC/FID.

Range: 4.4 - 180 ng
Correlation coefficient: 0.999
Slope: 4.9×10^{-3}
y-Intercept: -6.1×10^{-3}

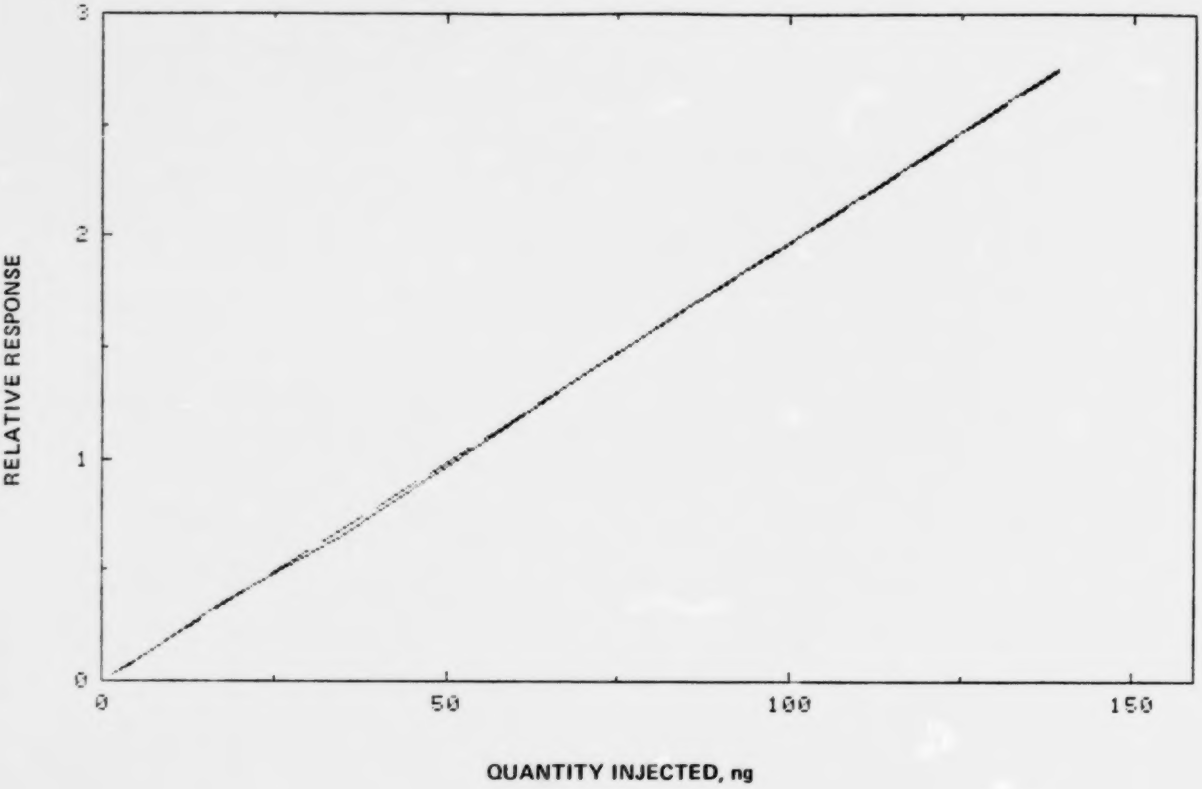

Figure C-43. Calibration curve for the determination of phosphorothioic acid, O,O-dimethyl-, O-(4-(methylthio)-m-tolyl) ester by GC/FID.

Range: 1.7 - 140 ng
Correlation coefficient: 0.999
Slope: 2.0×10^{-2}
y-Intercept: -9.7×10^{-3}

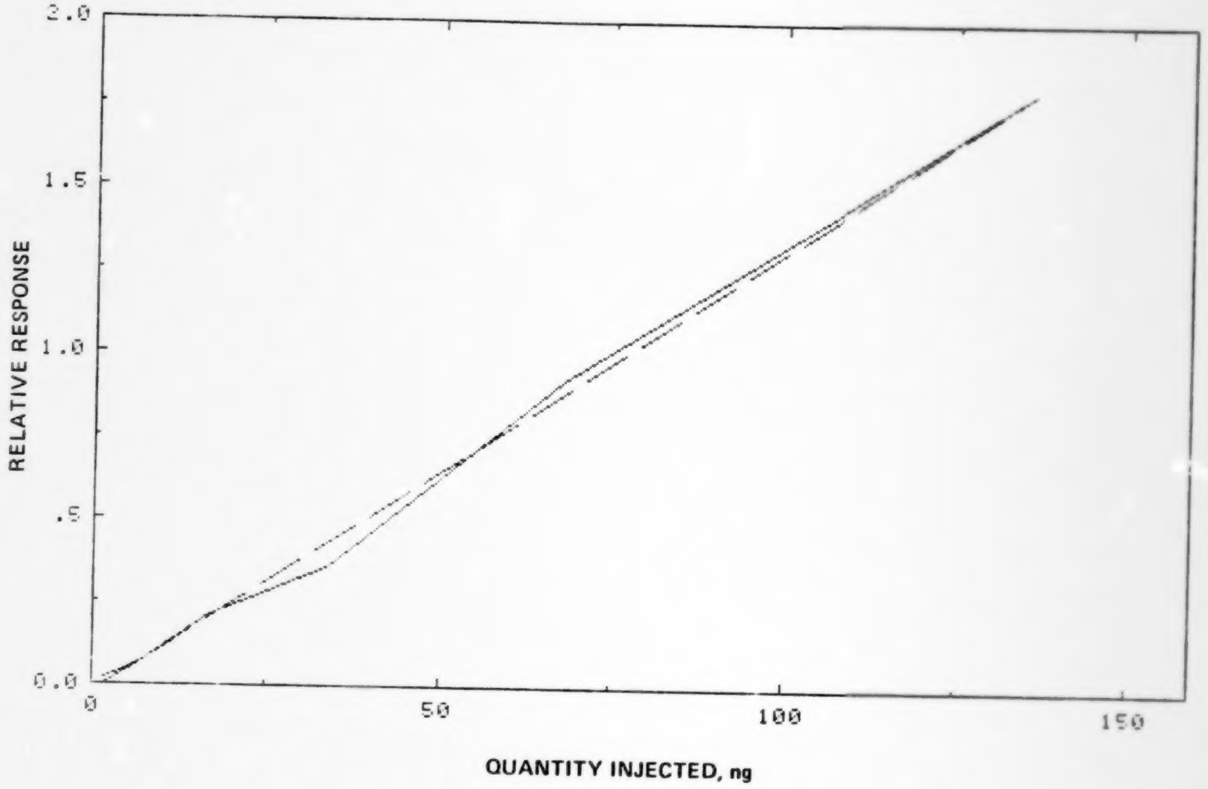

Figure C-44. Calibration curve for the determination of phosphorothioic acid, O,O-diethyl O-(p-(methylsulfinyl) phenyl) ester by GC/FID.

Range: 6.8 - 140 ng
Correlation coefficient: 0.998
Slope: 1.3×10^{-2}
y-Intercept: -1.9×10^{-2}

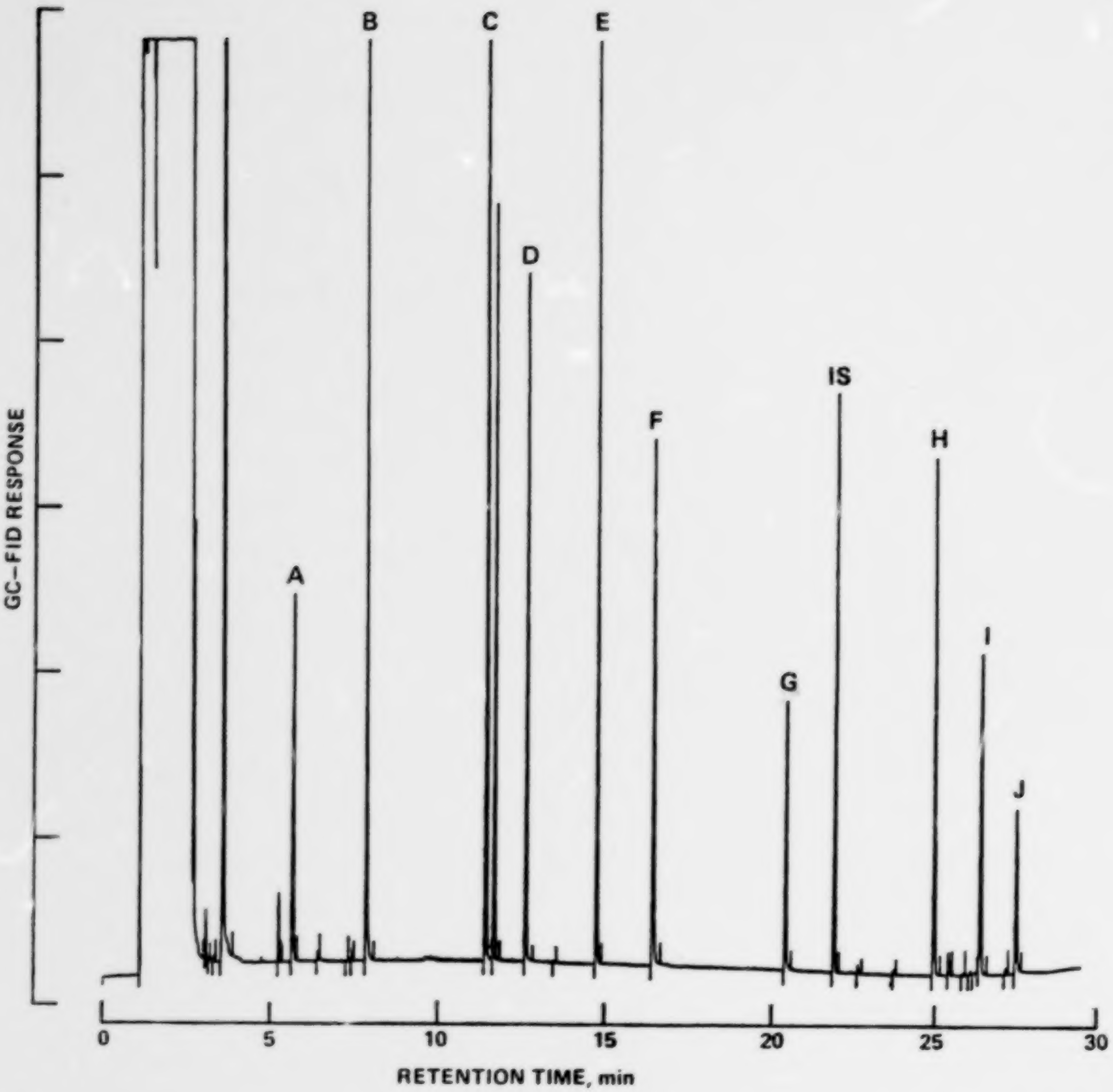

Figure C-45. Chromatogram by GC/FID for

(A) propionitrile, 2-hydroxy- (t_R = 5.8 min)
(B) styrene(t_R = 8.0 min)
(C) o-toluidine(t_R = 11.6 min)
(D) 2-oxetanone (2-oxepanone) (t_R = 12.8 min)
(E) o-anisidine, 5-methyl- (t_R = 14.9 min)
(F) toluene, 2,4-diamino- (t_R = 16.6 min)
(G) benzonitrile, 3,5-dibromo-4-hydroxy- (t_R = 20.6 min)
(H) aniline, p-(phenylazo)- (t_R = 25.2 min)
(I) 3-amino-9-ethyl carbazole(t_R = 26.5 min)
(J) pyridine, 2,6-diamino-3-(phenylazo)-,
monohydrochloride(t_R = 27.6 min)

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

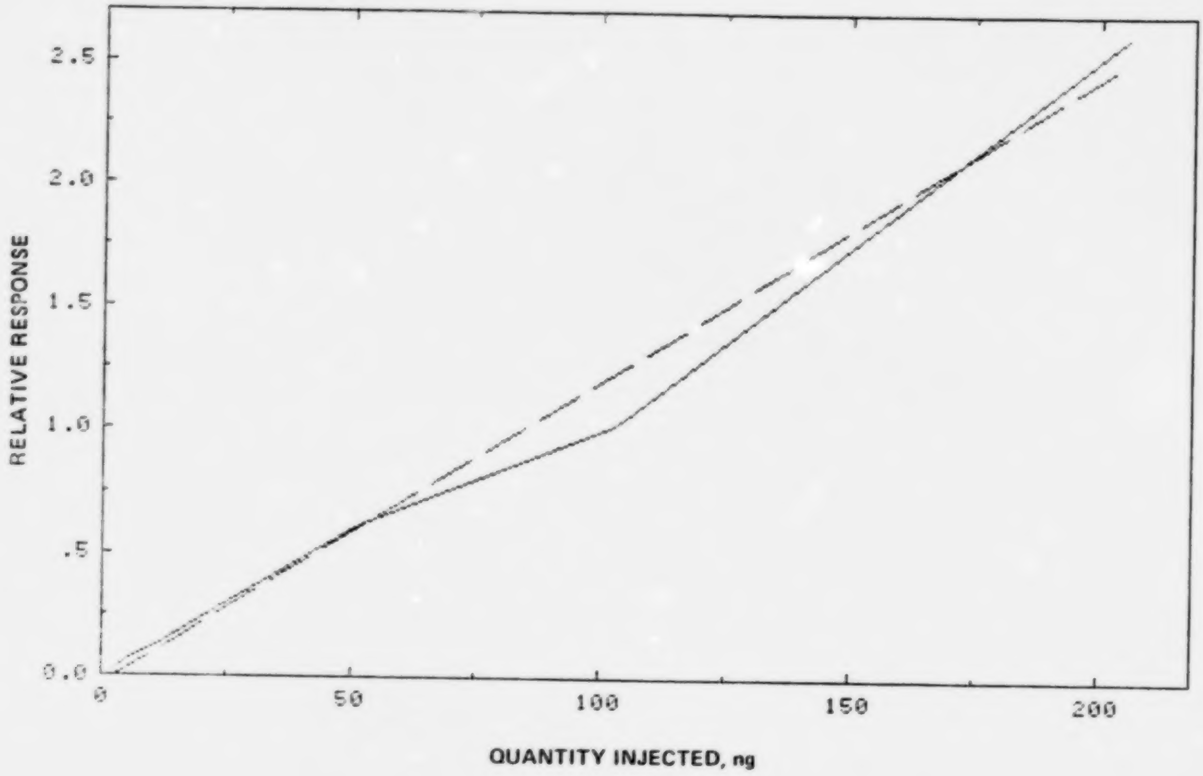

Figure C-46. Calibration curve for the determination of propionitrile, 2-hydroxy- by GC/FID.

Range: 10 - 210 ng
Correlation coefficient: 0.994
Slope: 1.2×10^{-2}
y-Intercept: -3.4×10^{-2}

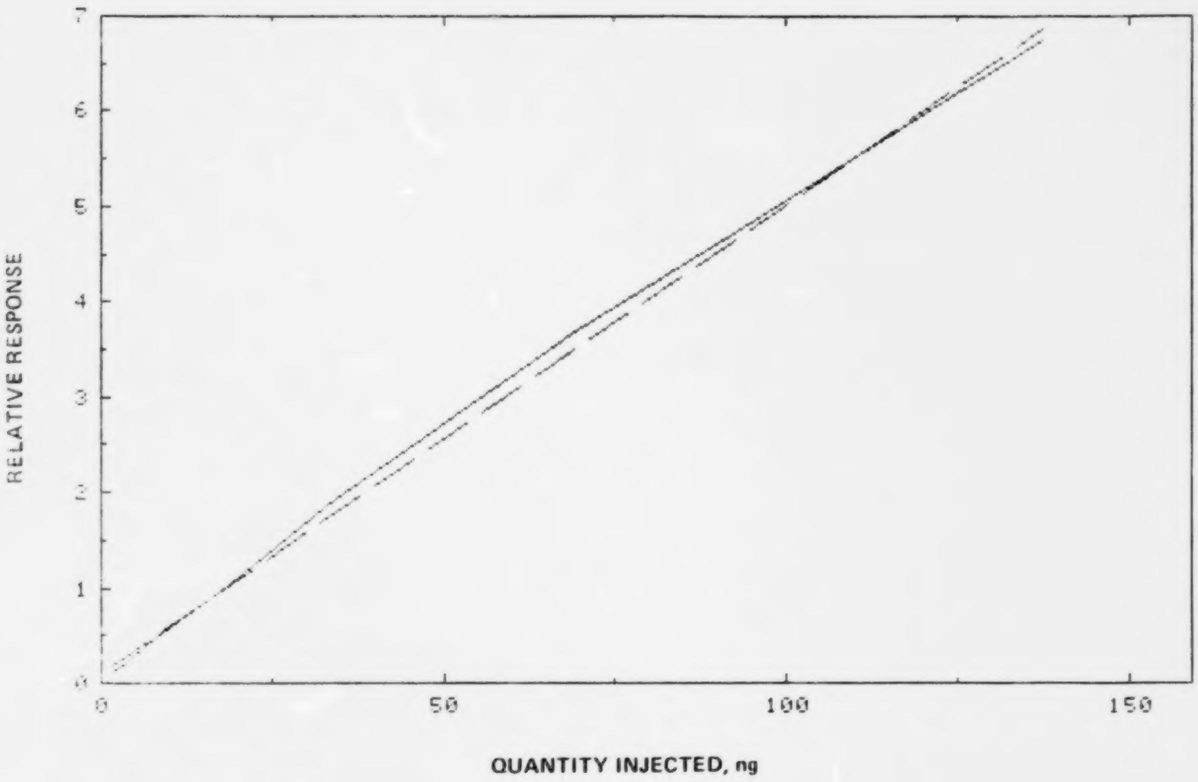

Figure C-47. Calibration curve for the determination of styrene by GC/FID.

Range: 6.9 - 140 ng
Correlation coefficient: 0.999
Slope: 4.9×10^{-2}
y-Intercept: 1.1×10^{-1}

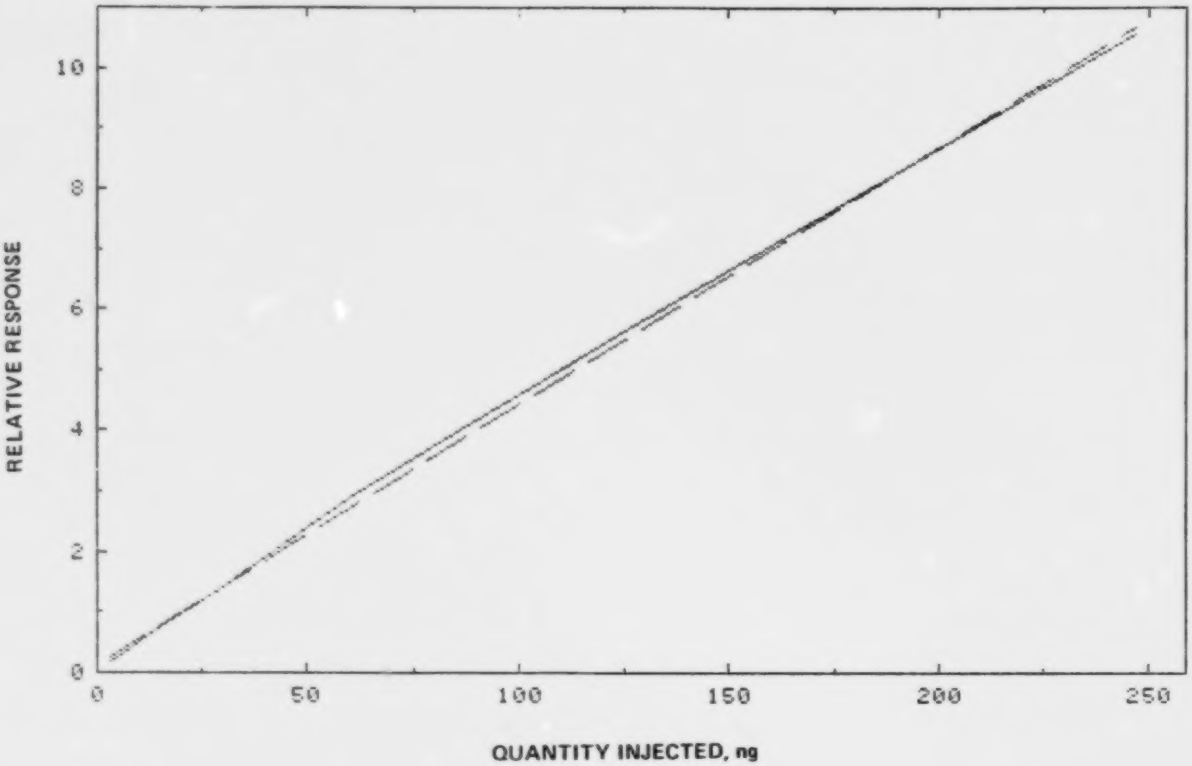

Figure C-48. Calibration curve for the determination of o-toluidine by GC/FID.

Range: 6.2 - 250 ng
Correlation coefficient: 0.999
Slope: 4.3×10^{-2}
y-Intercept: 1.4×10^{-1}

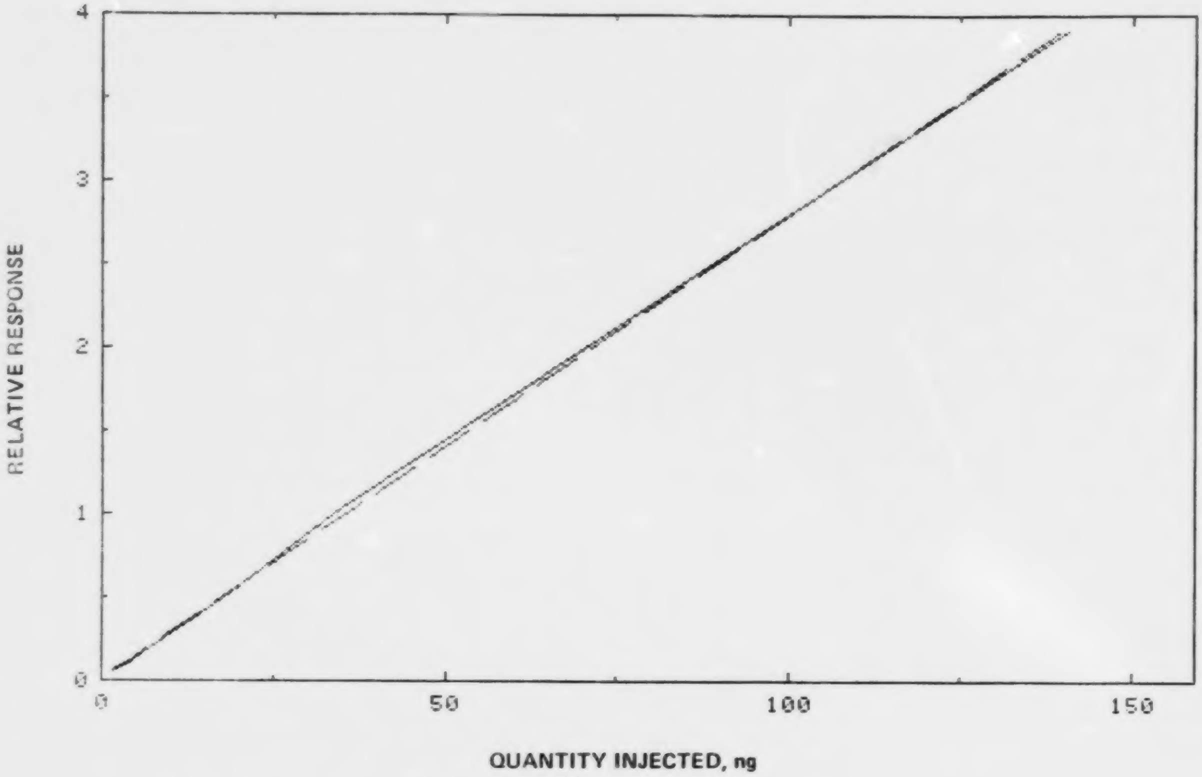

Figure C-49. Calibration curve for the determination of 2-oxepanone by GC/FID.

Range: 1.8 - 140 ng
Correlation coefficient: 0.999
Slope: 2.8×10^{-2}
y-Intercept: 1.0×10^{-2}

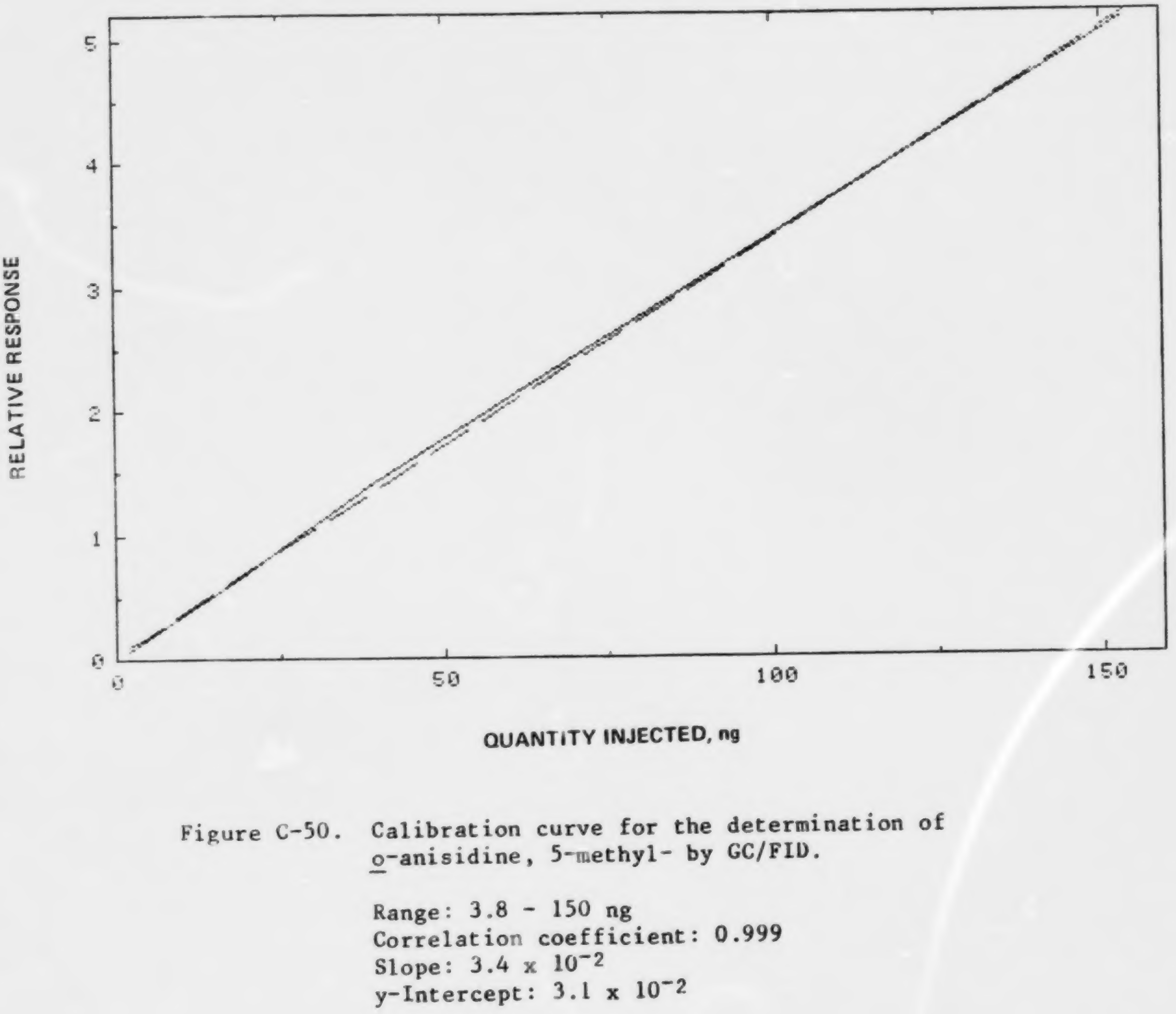

Figure C-50. Calibration curve for the determination of o-anisidine, 5-methyl- by GC/FID.

Range: 3.8 - 150 ng
Correlation coefficient: 0.999
Slope: 3.4×10^{-2}
y-Intercept: 3.1×10^{-2}

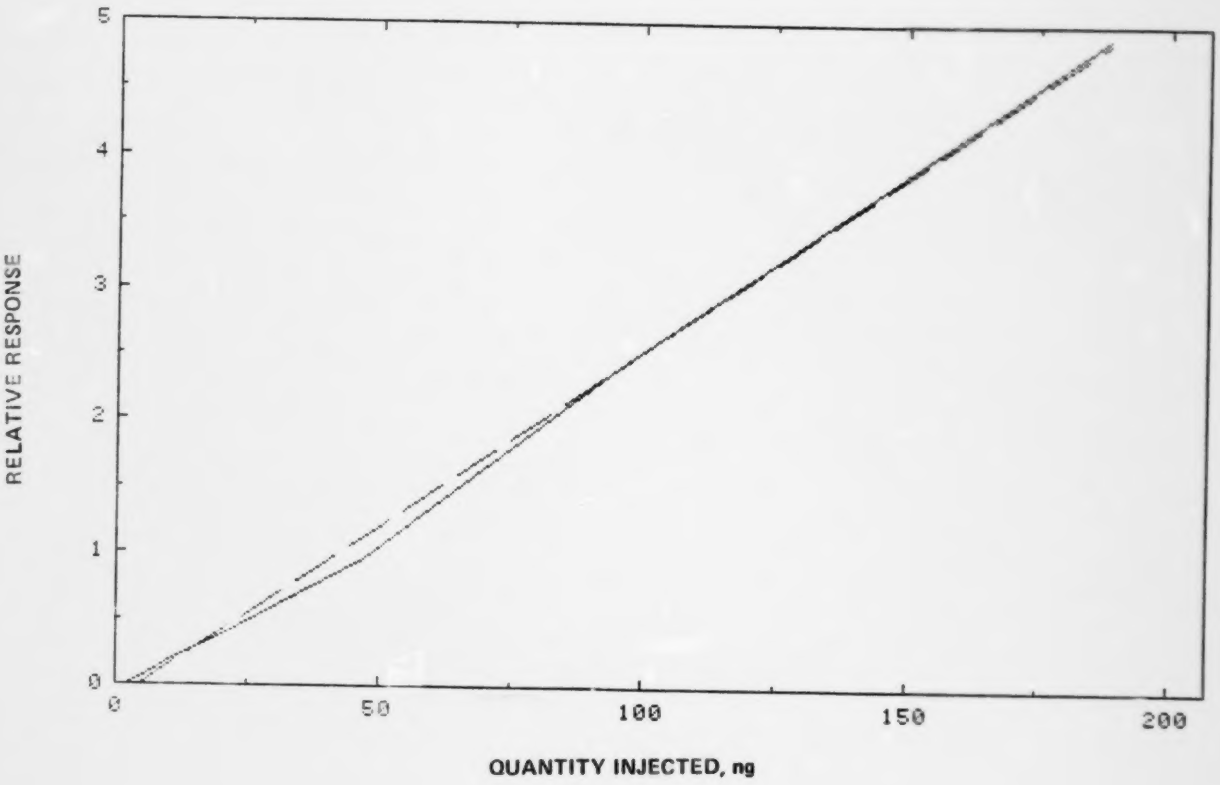

Figure C-51. Calibration curve for the determination of toluene, 2,4-diamino- by GC/FID.

Range: 9.4 - 190 ng
Correlation coefficient: 0.999
Slope: 2.6×10^{-2}
y-Intercept: -1.2×10^{-1}

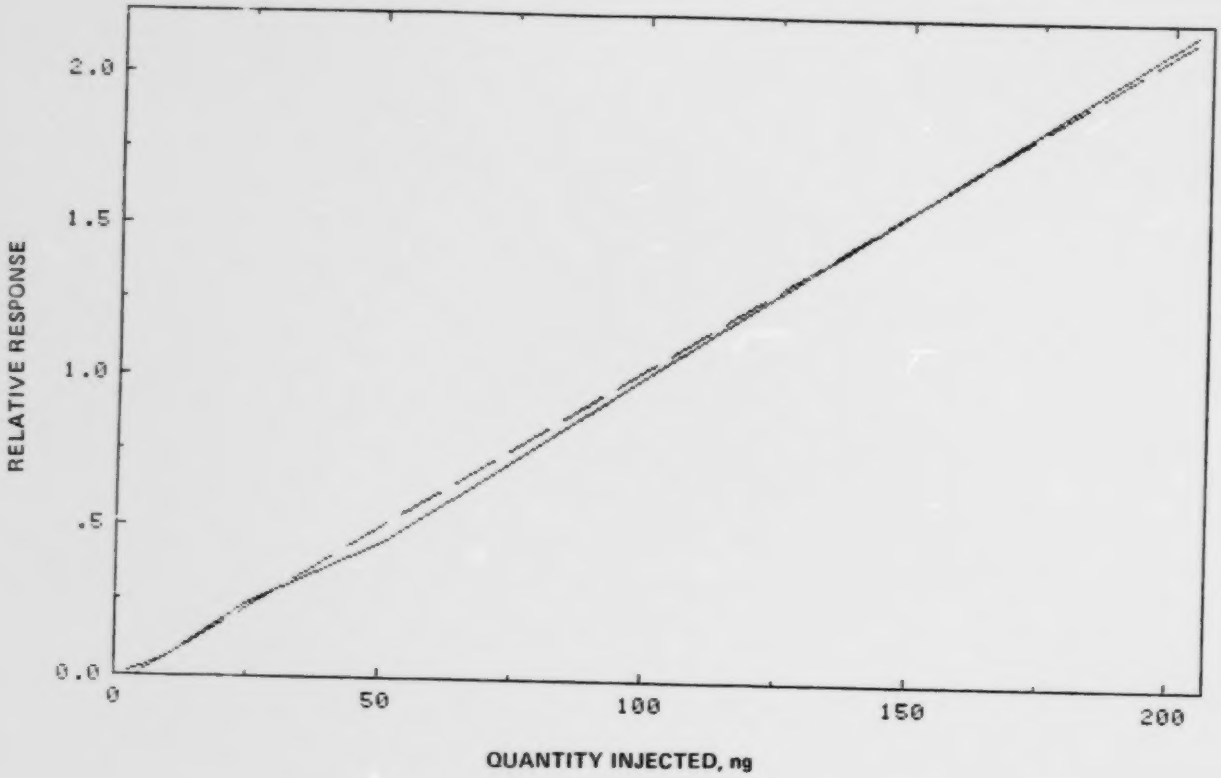

Figure C-52. Calibration curve for the determination of benzonitrile, 3,5-dibromo-4-hydroxy- by GC/FID.

Range: 10 - 200 ng
Correlation coefficient: 0.999
Slope: 1.1×10^{-2}
y-Intercept: -4.2×10^{-2}

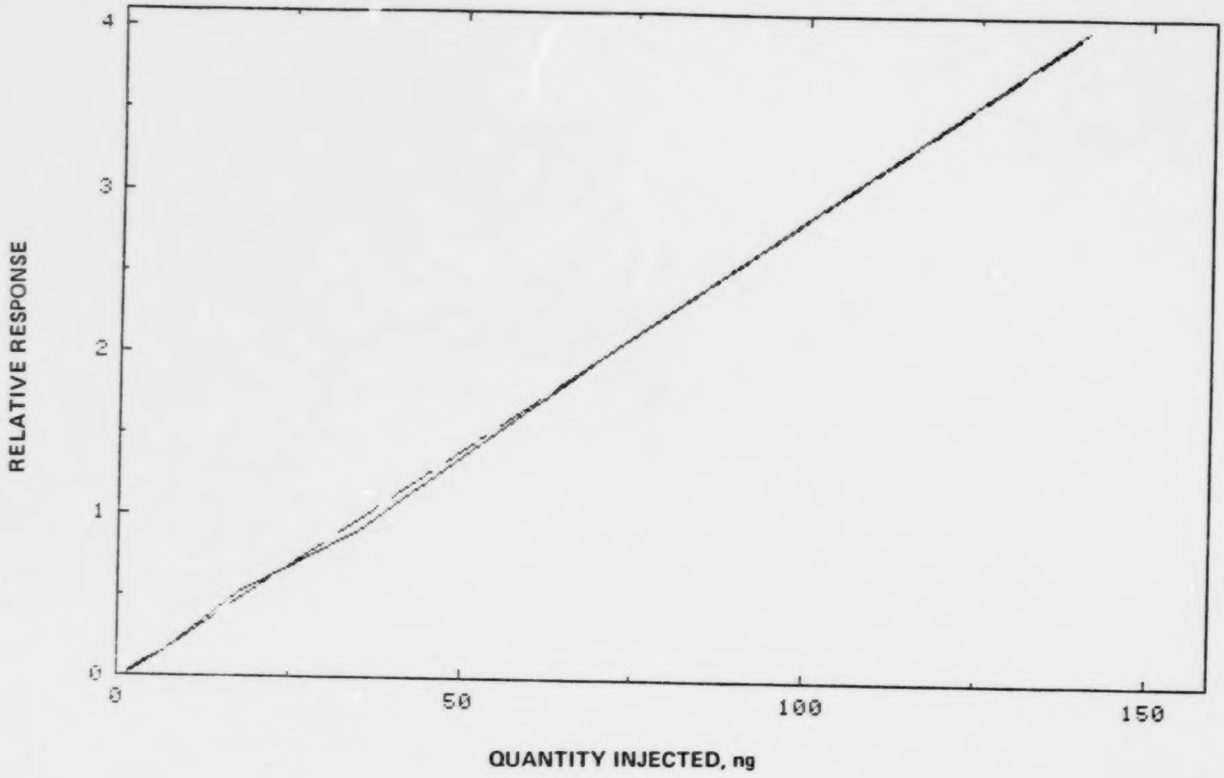

Figure C-53. Calibration curve for the determination of aniline, p-(phenylazo)- by GC/FID.

Range: 7.0 - 140 ng
Correlation coefficient: 0.999
Slope: 2.9 x 10^{-2}
y-Intercept: -3.7 x 10^{-2}

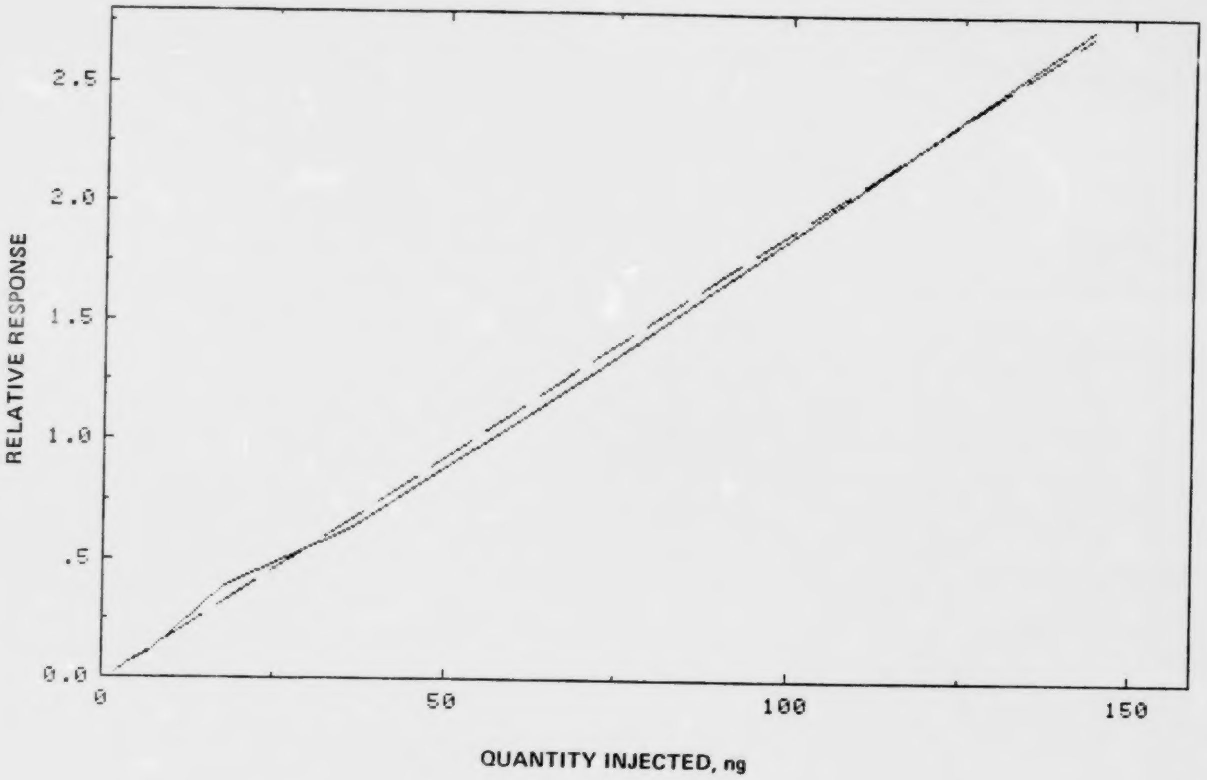

Figure C-54. Calibration curve for the determination of 3-amino-9-ethyl carbazole by GC/FID.

Range: 1.8 - 140 ng
Correlation coefficient: 0.999
Slope: 1.9×10^{-2}
y-Intercept: -1.7×10^{-2}

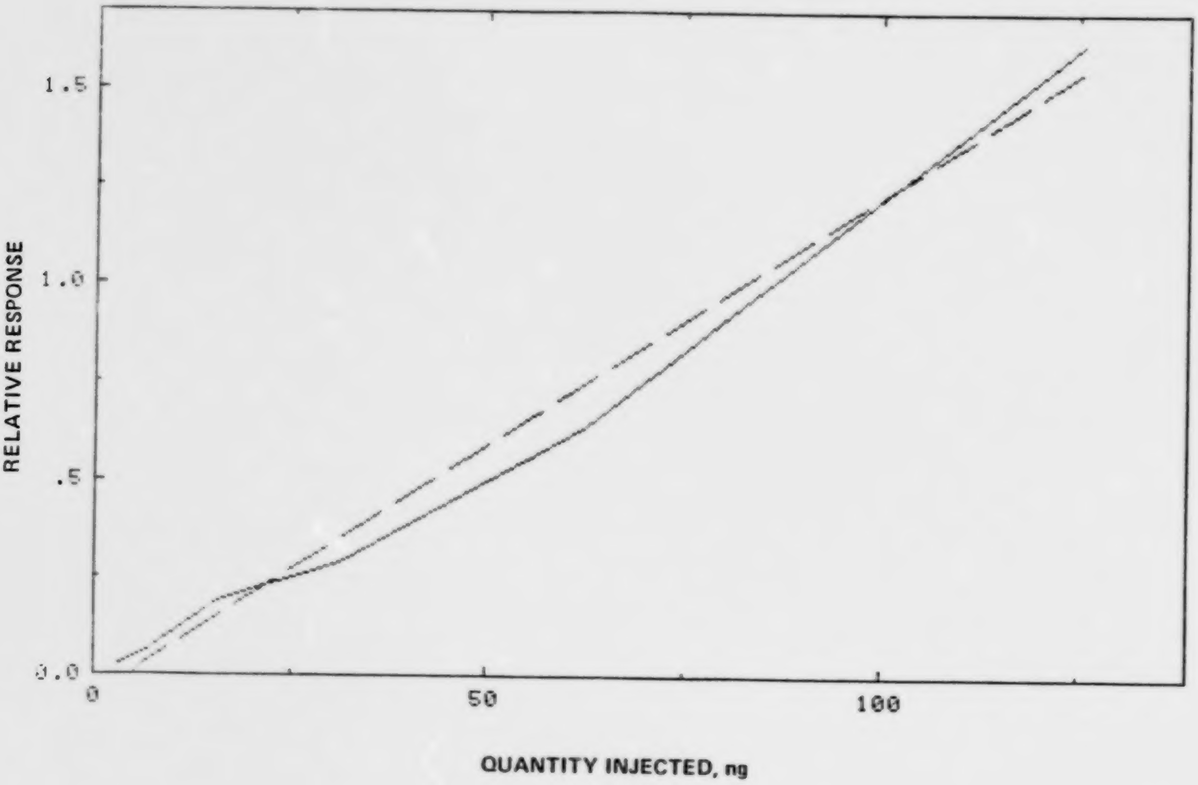

Figure C-55. Calibration curve for the determination of pyridine, 2,6-diamino-3-(phenylazo)-, monohydrochloride by GC/FID.

Range: 16 - 130 ng
Correlation coefficient: 0.993
Slope: 1.3×10^{-2}
y-Intercept: -5.3×10^{-2}

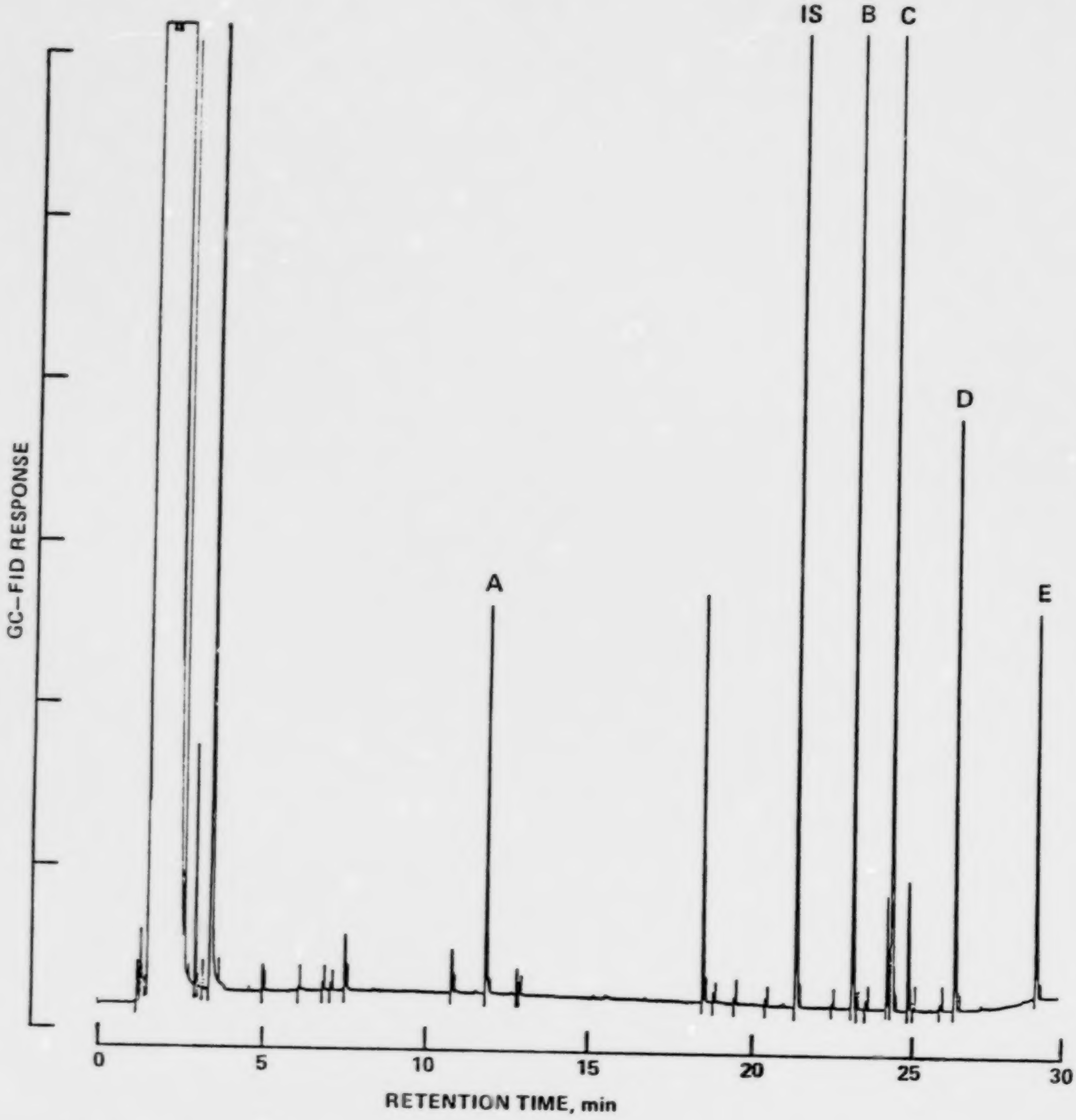

Figure C-56. Chromatogram by GC/FID for

(A) tetraethylpyrophosphate(t_R = 12.5 min),
(B) succinic acid, mercapto-, diethyl ester, S-ester with O,O-dimethyl phosphorodithioate(t_R = 23.0 min),
(C) crotonic acid, 3-hydroxy-, alpha-methylbenzyl ester, dimethyl phosphate, (E)- (t_R = 24.2 min),
(D) phosphorodithioic acid, S,S'-methylene O,O,O',O'-tetraethyl ester(t_R = 26.5 min), and
(E) phosphorodithioic acid, O,O-dimethyl ester, S-ester with 3-(mercaptomethyl)-1,2,3-benzotriazin-4(3H)-one(t_R = 29.1 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

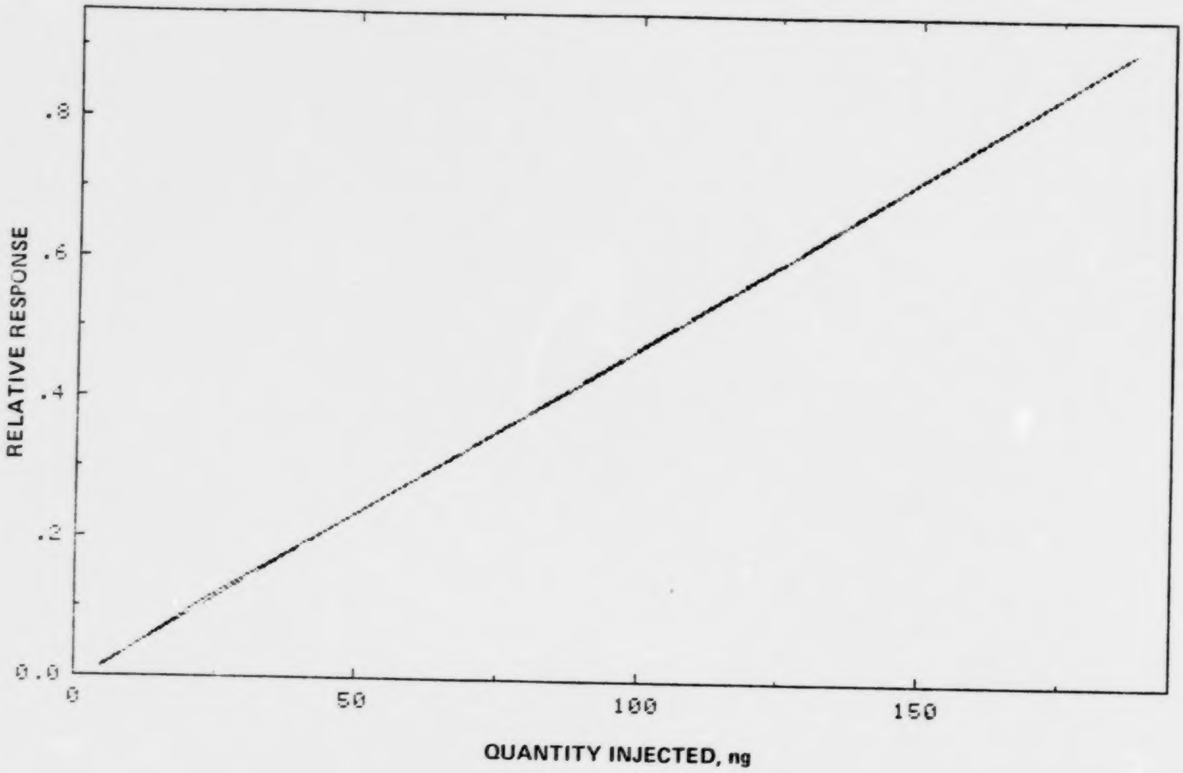

Figure C-57. Calibration curve for the determination of tetraethylpyrophosphate by GC/FID.

Range: 4.7 - 190 ng
Correlation coefficient: 0.999
Slope: 4.8×10^{-3}
y-Intercept: -6.8×10^{-3}

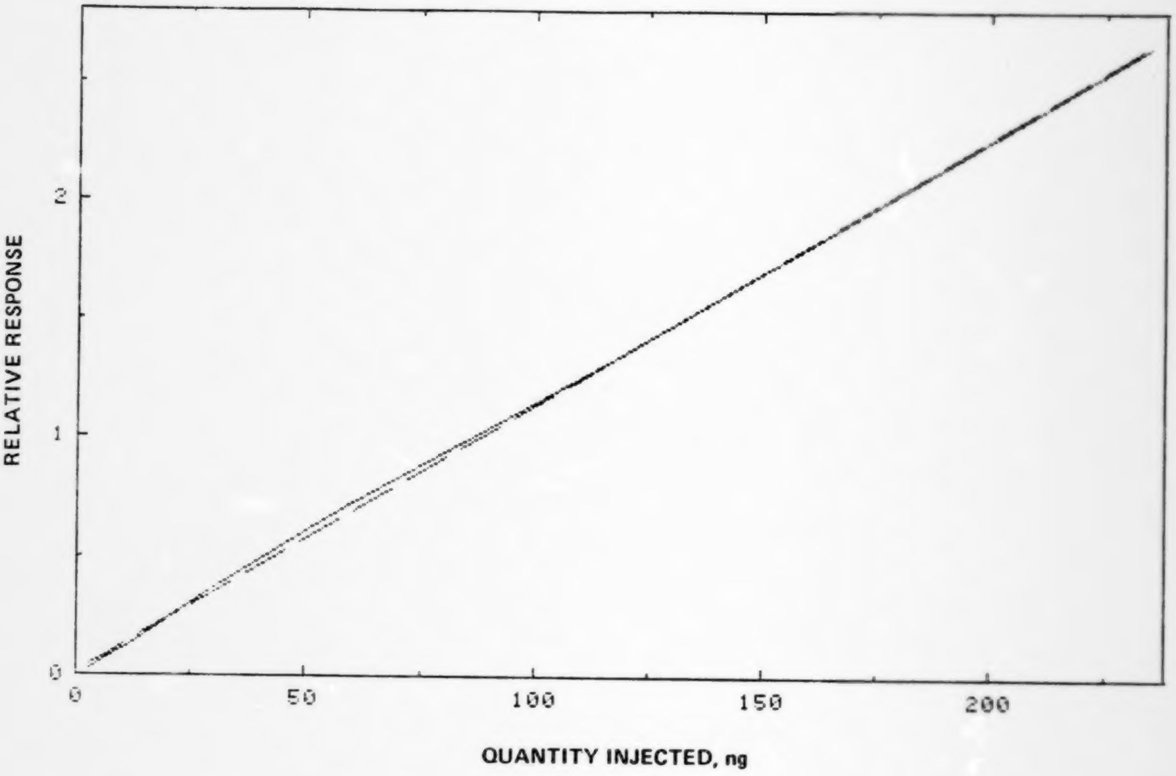

Figure C-58. Calibration curve for the determination of succinic acid, mercapto-, diethyl ester, S-ester with O,O-dimethyl phosphorodithioate by GC/FID.

Range: 29 - 240 ng
Correlation coefficient: 0.999
Slope: 1.1×10^{-2}
y-Intercept: 1.1×10^{-2}

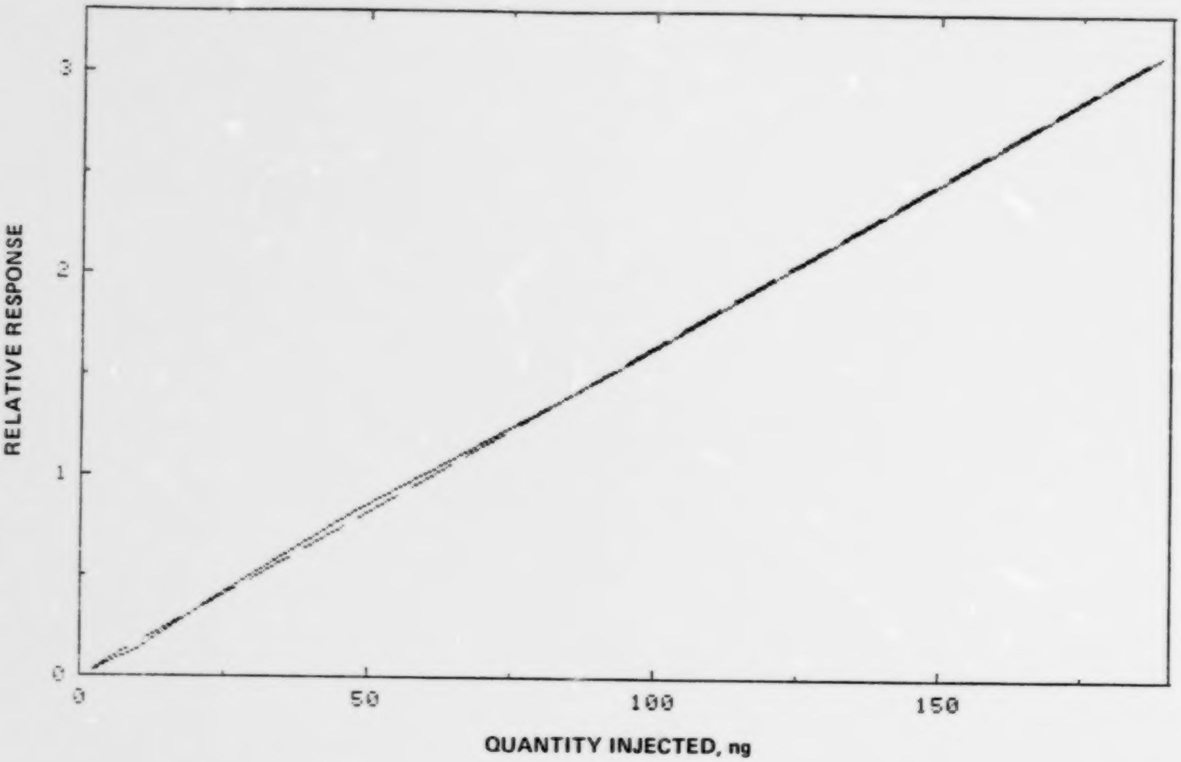

Figure C-59. Calibration curve for the determination of crotonic acid, 3-hydroxy-, alpha-methylbenzyl ester, dimethyl phosphate, (E)- by GC/FID.

Range: 2.4 - 190 ng
Correlation coefficient: 0.999
Slope: 1.6×10^{-2}
y-Intercept: -7.2×10^{-3}

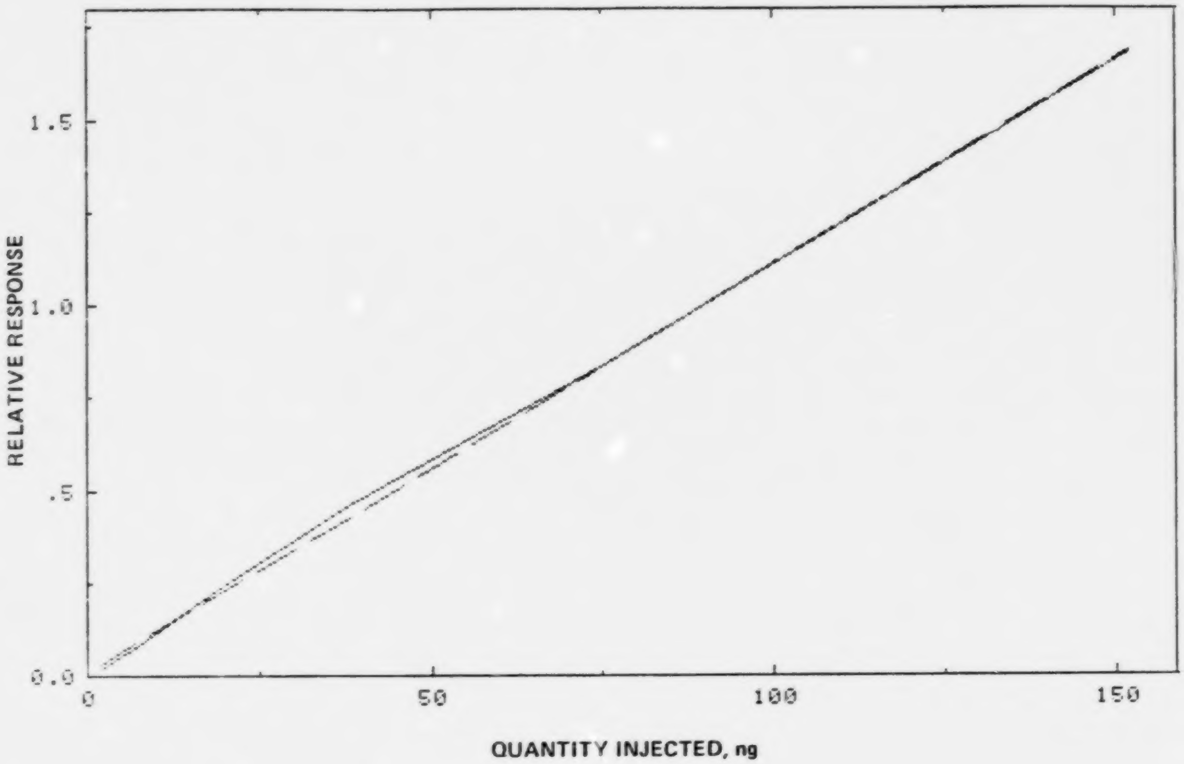

Figure C-60. Calibration curve for the determination of phosphorodithioic acid, S,S'-methylene O,O,O',O'-tetraethyl ester by GC/FID.

Range: 1.9 - 150 ng
Correlation coefficient: 0.999
Slope: 1.1×10^{-2}
y-Intercept: 8.5×10^{-3}

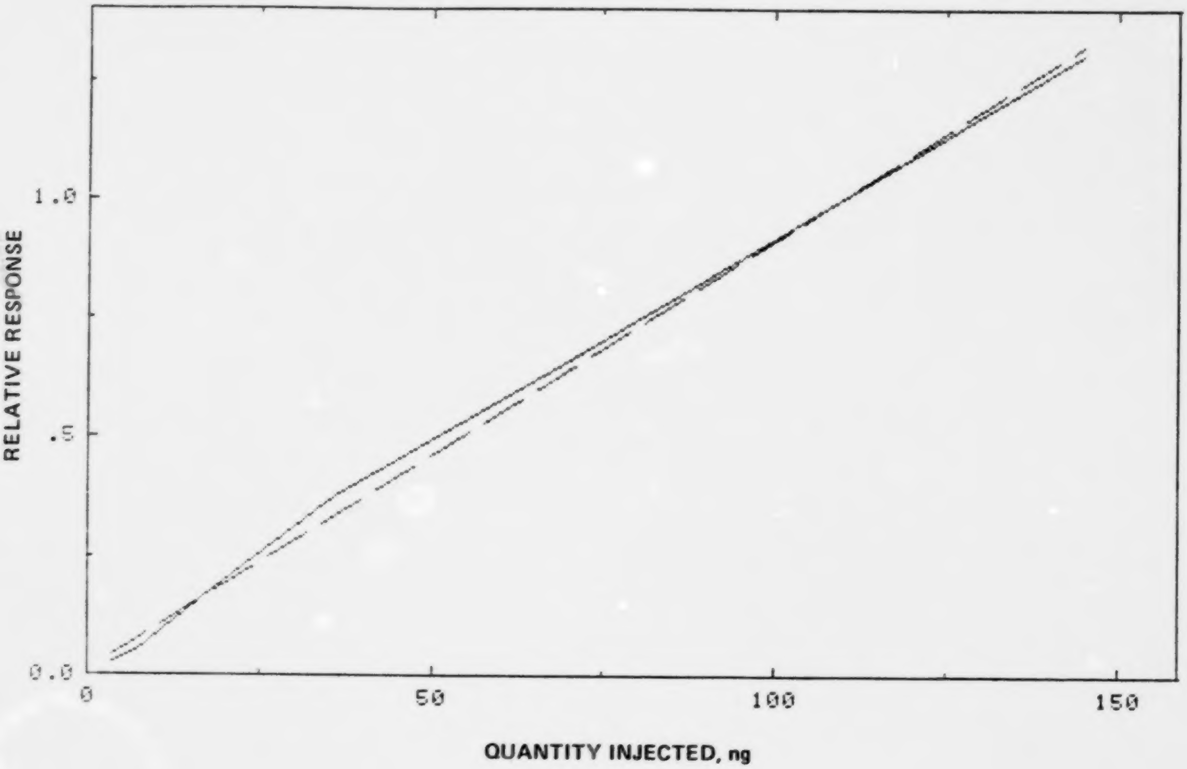

Figure C-61. Calibration curve for the determination of phosphorodithioic acid, O,O-dimethyl ester, S-ester with 3-(mercaptomethyl)-1,2,3-benzotriazin-4(3H)-one by GC/FID.

Range: 18 - 140 ng
Correlation coefficient: 0.999
Slope: 9.1×10^{-3}
y-Intercept: 9.9×10^{-3}

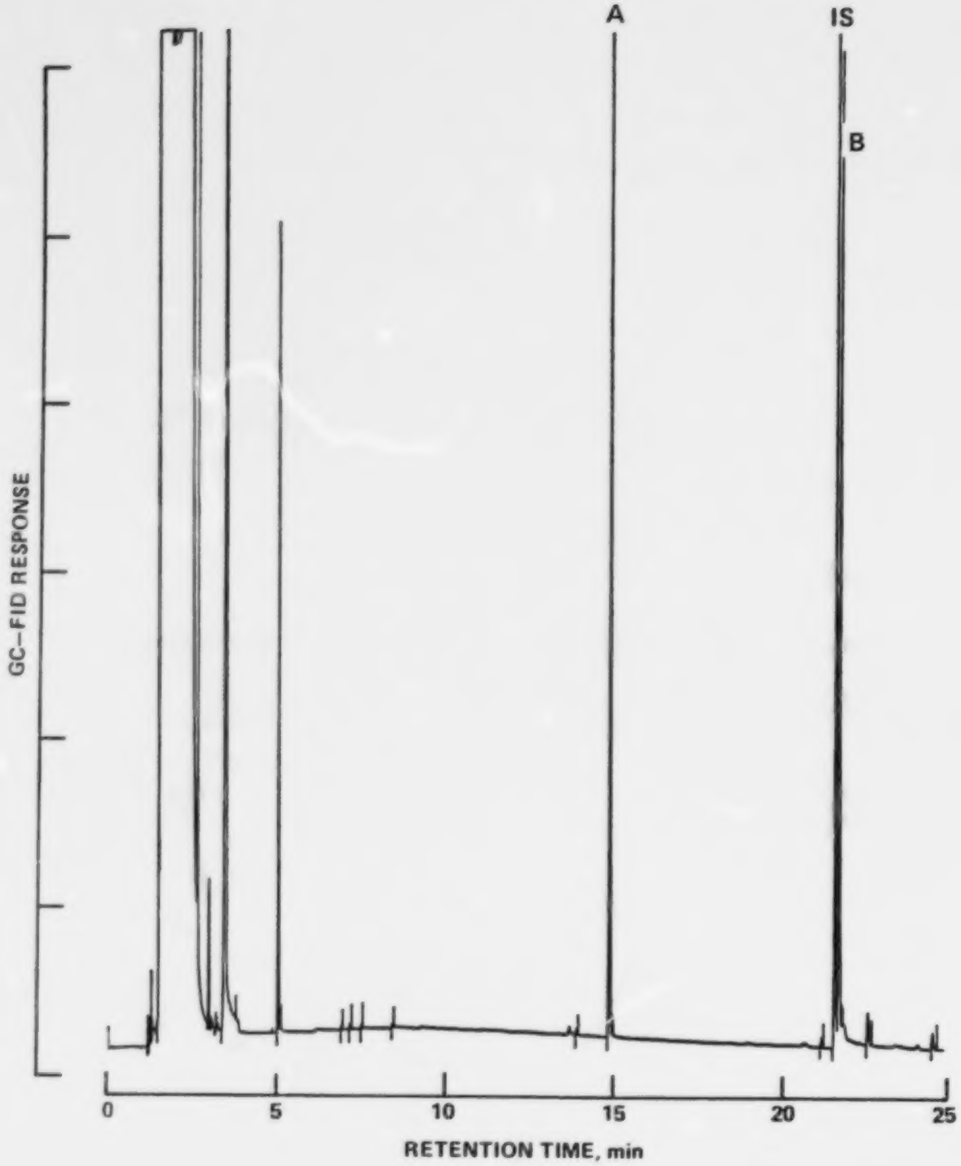

Figure C-62. Chromatogram by GC/FID for

(A) o-toluidine, 5-chloro- (t_R = 15.0 min) and
(B) 1,4-naphthoquinone, 2,3-dichloro- (t_R = 21.8 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

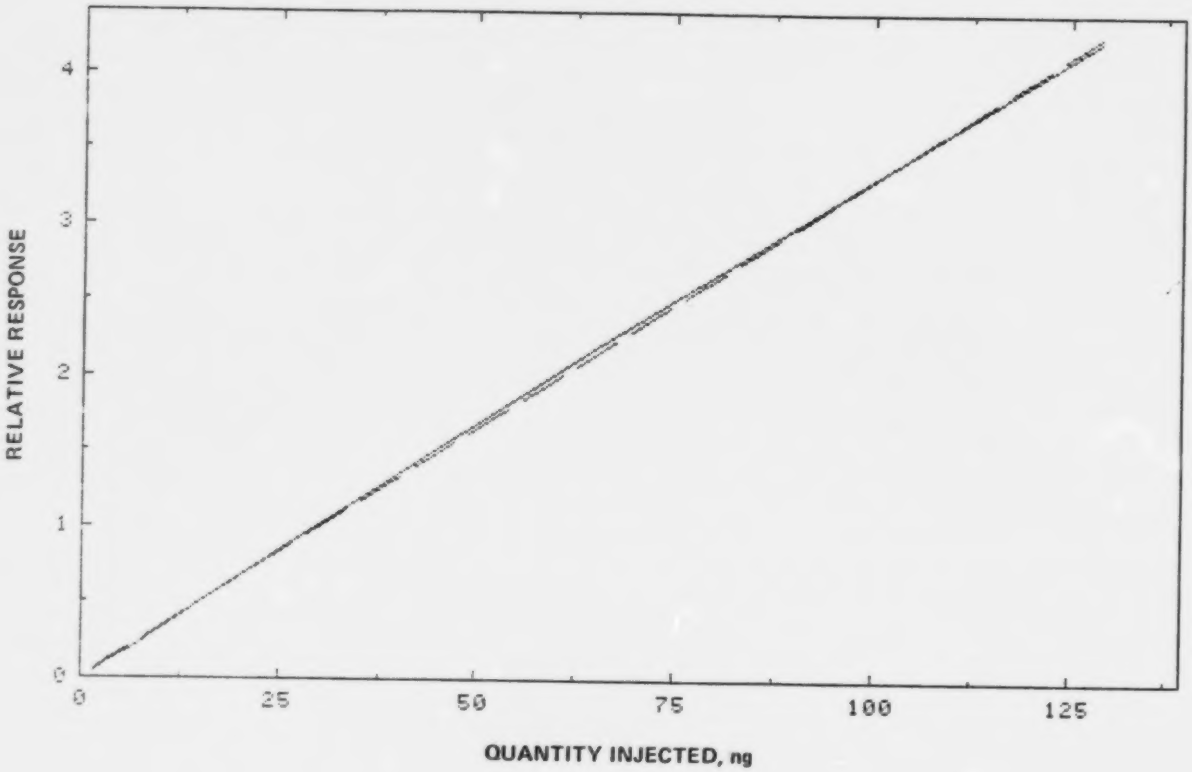

Figure C-63. Calibration curve for the determination of o-toluidine, 5-chloro- by GC/FID.

Range: 3.0 - 130 ng
Correlation coefficient: 0.999
Slope: 3.3×10^{-2}
y-Intercept: 7.7×10^{-3}

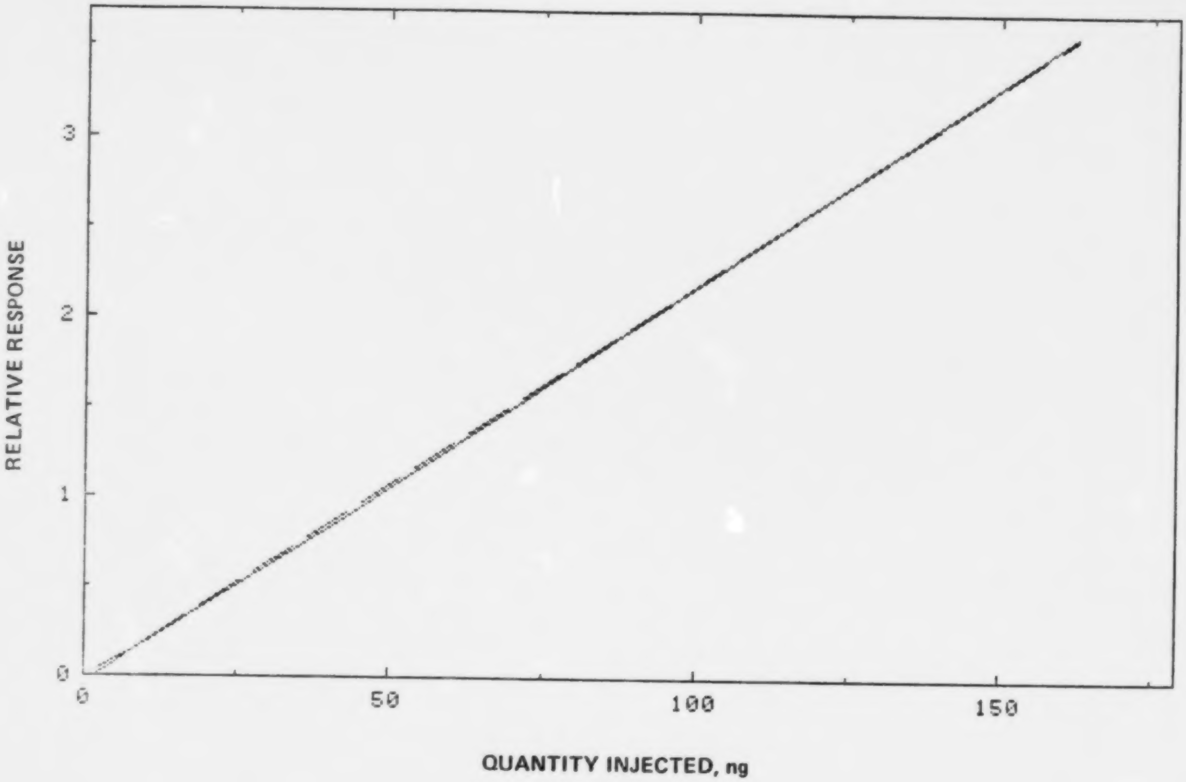

Figure C-64. Calibration curve for the determination of 1,4-naphthoquinone, 2,3-dichloro- by GC/FID.

Range: 4.0 - 160 ng
Correlation coefficient: 0.999
Slope: 2.2×10^{-2}
y-Intercept: -3.7×10^{-2}

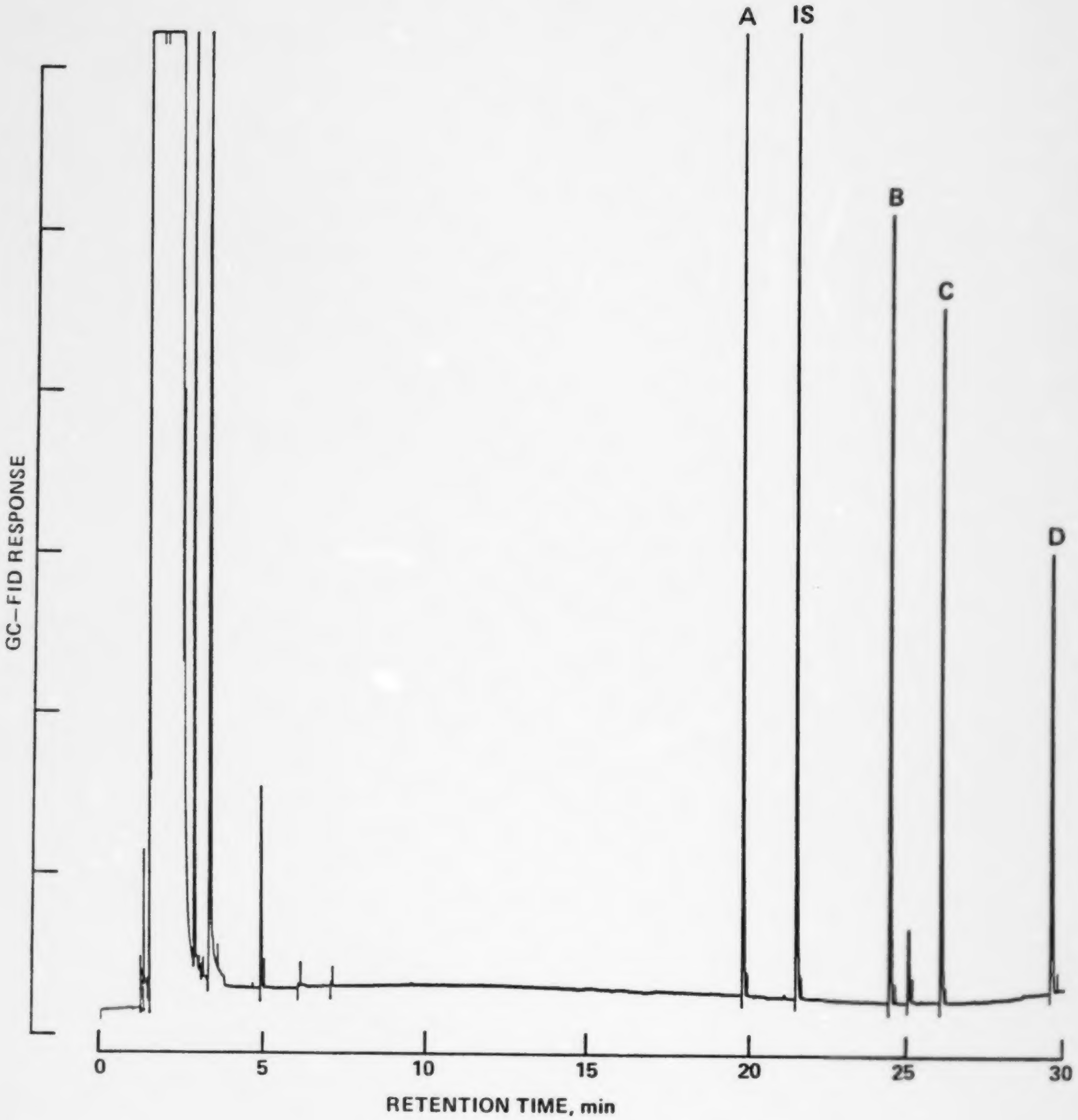

Figure C-65. Chromatogram by GC/FID for

(A) p-toluidine, alpha,alpha,alpha-trifluoro-2,6-dinitro-N,N-dipropyl- (t_R = 20.0 min),
(B) heptachlor epoxide(t_R = 24.6 min),
(C) ether, 2,4-dichlorophenyl p-nitrophenyl(t_R = 26.2 min), and
(D) 1,3,4-metheno-1H-cyclobuta[cd]pentalene, 1,1a,2,2,3,3a,4,5,5,5a,5b,6- dodecachloro-octahydro- (t_R = 29.6 min)

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

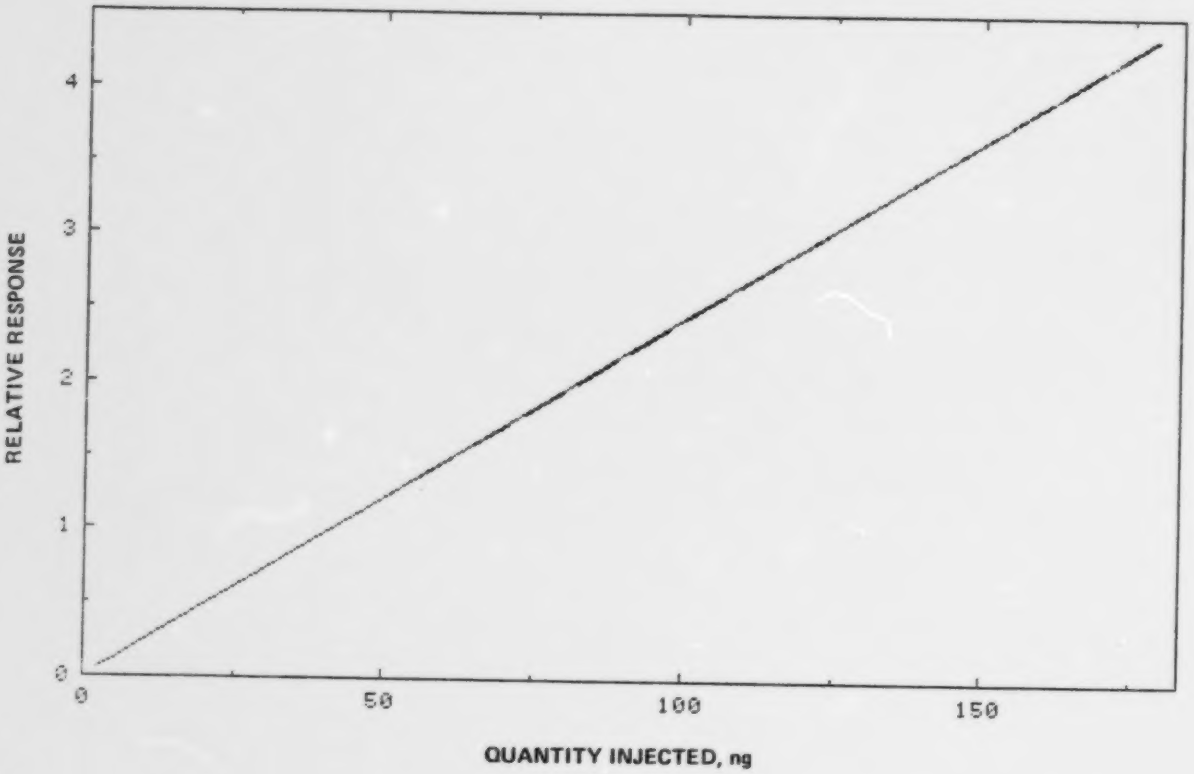

Figure C-66. Calibration curve for the determination of p-toluidine, alpha,alpha,alpha-trifluoro-2,6-dinitro-N,N-dipropyl- by GC/FID.

Range: 2.2 - 180 ng
Correlation coefficient: 0.999
Slope: 2.4×10^{-2}
y-Intercept: 4.6×10^{-5}

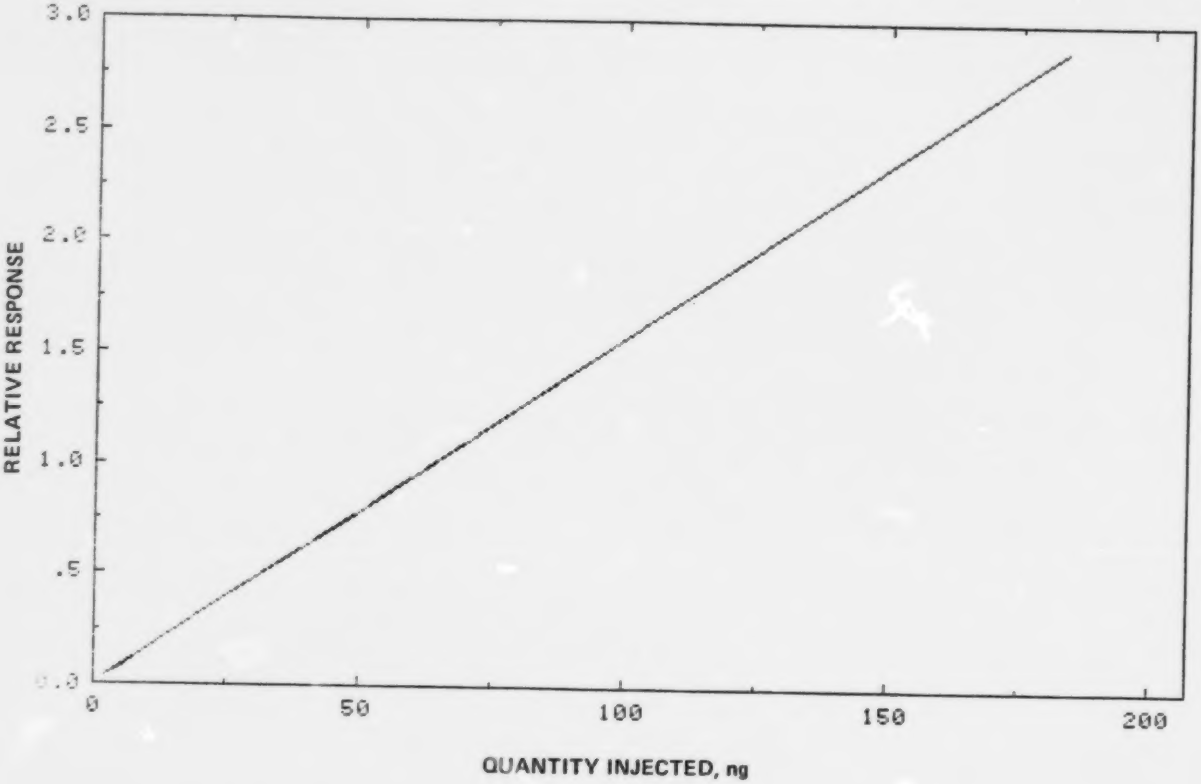

Figure C-67. Calibration curve for the determination of heptachlor epoxide by GC/FID.

Range: 2.3 - 180 ng
Correlation coefficient: 0.999
Slope: 1.6×10^{-2}
y-Intercept: 5.0×10^{-4}

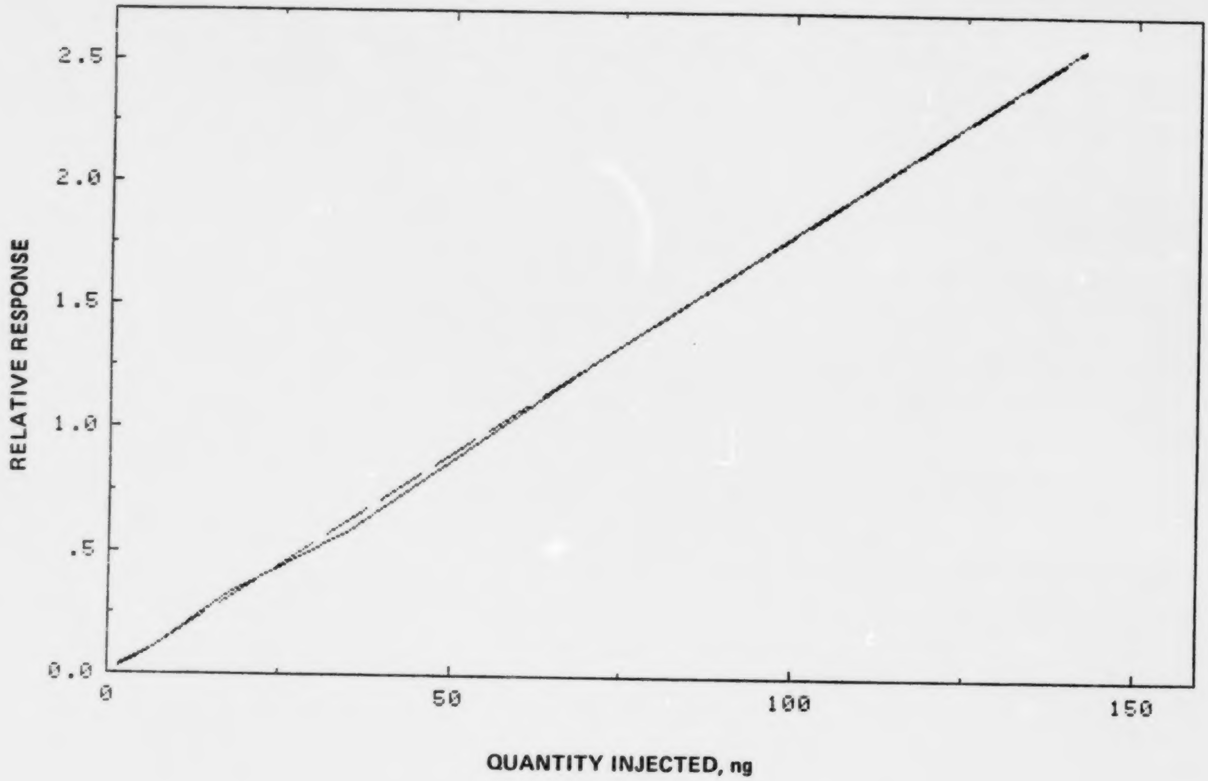

Figure C-68. Calibration curve for the determination of ether, 2,4-dichlorophenyl p-nitrophenyl by GC/FID.

Range: 1.8 - 140 ng
Correlation coefficient: 0.999
Slope: 1.8×10^{-2}
y-Intercept: -8.6×10^{-3}

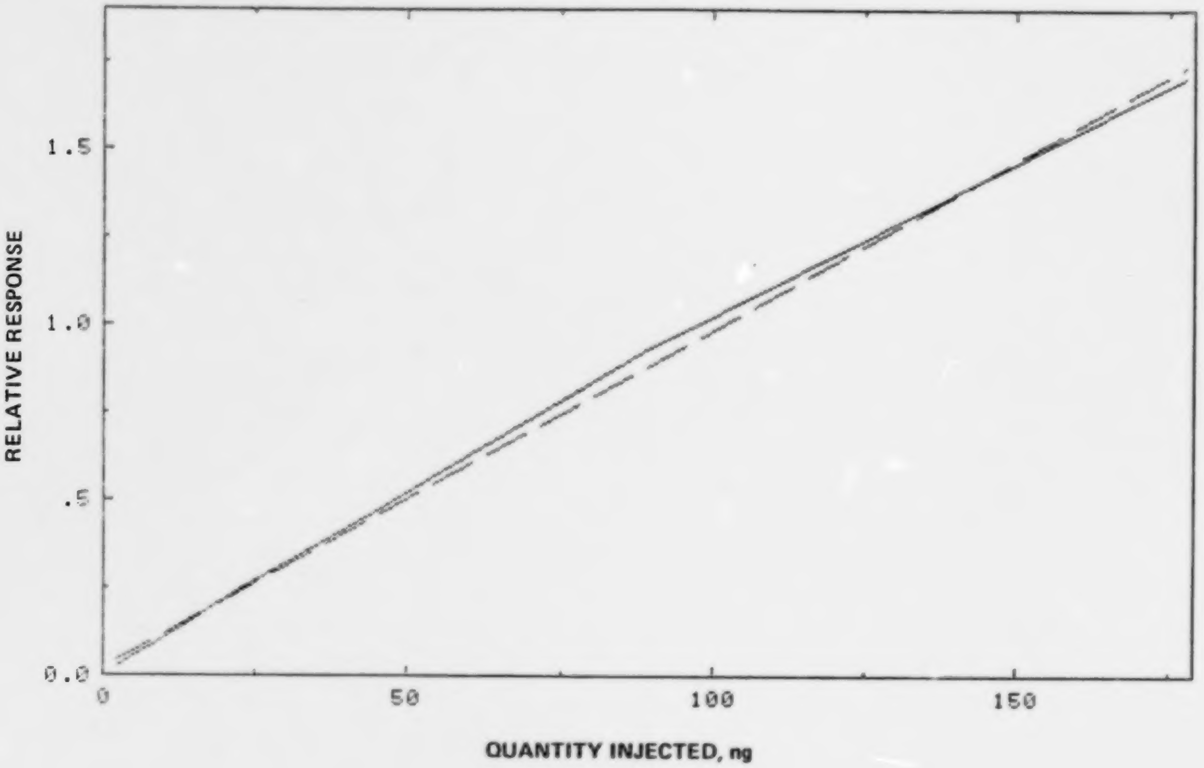

Figure C-69. Calibration curve for the determination of 1,3,4-metheno-1H-cyclobuta[cd]pentalene, 1,1a,2,2,3,3a,4,5,5,5a,5b,6- dodecachlorooctahydro- by GC/FID.

Range: 8.9 - 180 ng
Correlation coefficient: 0.999
Slope: 9.6×10^{-3}
y-Intercept: 2.0×10^{-2}

APPENDIX D

CHROMATOGRAMS, MASS SPECTRA, AND CALIBRATION CURVES FOR GC/MS

Index

Compound	Reference chromatogram Figure No.	Reference mass spectrum Figure No.	Reference calibration curve Figure No.
3-Amino-9-ethyl carbazole	D-89	D-104	D-105
Aniline, 4,4'-methylenebis(N,N-dimethyl-	D-1	D-8	D-9
Aniline, p-(phenylazo)-	D-89	D-102	D-103
Aniline, 2,4,5-trimethyl-	D-51	D-56	D-57
o-Anisidine	D-51	D-54	D-55
o-Anisidine hydrochloride	D-80	D-83	D-84
o-Anisidine, 5-methyl-	D-89	D-96	D-97
o-Anisidine, 5-nitro-	D-51	D-58	D-59
Benzonitrile, 3,5-dibromo-4-hydroxy-	D-89	D-100	D-101
Biphenyl, 4-nitro	D-51	D-60	D-61
Carbamic acid, methyl-, 2,3-dihydro-2,2-dimethyl-7-benzofuranyl ester	D-1	D-2	D-3
Carbamic acid, methyl-, 4-dimethylamino-3,4-xylyl ester	D-10	D-11	D-12
Carbamic acid, methyl-, 2,3-(dimethylmethylenedioxy)phenyl ester	D-13	D-14	D-15
Carbamic acid, methyl-, 1-naphthyl ester	D-1	D-4	D-5
Carbanilic acid, m-chloro-, 4-chloro-2-butynyl ester	D-13	D-18	D-19
Chlorobenzene	D-22	D-23	D-24
Coumarin, 3-chloro-7-hydroxy-4-methyl-, O-ester with O,O-diethyl phosphorothioate	D-38	D-49	D-50
Crotonic acid, 3-hydroxy-, alpha-methylbenzyl ester, dimethyl phosphate, (E)-	D-108	D-113	D-114
4-Cyclohexene-1,2-dicarboximide, N-((1,1,2,2-tetrachloroethyl)thio)	D-13	D-20	D-21
4-Cyclohexene-1,2-dicarboximide, N-(trichloromethyl)thio-	D-1	D-6	D-7
m-Dichlorobenzene	D-25	D-26	D-27
o-Dichlorobenzene	D-28	D-31	D-32
p-Dichlorobenzene	D-28	D-29	D-30
Ether, 2,4-dichlorophenyl p-nitrophenyl	D-127	D-132	D-133
Heptachlor epoxide	D-127	D-130	D-131
Hexabromobiphenyl	D-33	D-34	D-35
Hydroquinone	D-80	D-85	D-86

(continued)

APPENDIX D
(continued)

CHROMATOGRAMS, MASS SPECTRA, AND CALIBRATION CURVES FOR GC/MS

Index

Compound	Reference chromato-gram Figure No.	Reference mass spectrum Figure No.	Reference calibration curve Figure No.
Hydroxylamine, *N*-nitroso-*N*-phenyl-, ammonium salt	D-75	D-78	D-79
1,3,4-Metheno-1*H*-cyclobuta[cd]pentalene, 1,1a,2,2,3,3a,4,5,5,5a,5b,6-dodecachlorooctahydro-	D-33	D-36	D-37
1,4-Naphthoquinone, 2,3-dichloro-	D-108	D-122	D-123
2-Oxetanone (2-Oxepanone)	D-89	D-94	D-95
o-Phenylenediamine, 4-chloro-	D-80	D-87	D-88
Phosphonothioic acid, phenyl-, *O*-(4-bromo-2,5-dichloro-phenyl) *O*-methyl ester	D-38	D-47	D-48
Phosphoric acid, 2-chloro-1-(2,4-dichlorophenyl) vinyl diethyl ester	D-38	D-41	D-42
Phosphoric acid, 1,2-dibromo-2,2-dichloroethyl dimethyl ester	D-66	D-69	D-70
Phosphoric acid, dimethyl ester, ester with (E)-3-hydroxy-*N*,*N*-dimethylcrotonamide	D-38	D-39	D-40
Phosphoric acid, trimethyl ester	D-51	D-52	D-53
Phosphoric acid, tri-*o*-tolyl ester	D-51	D-64	D-65
Phosphoric triamide, hexamethyl-	D-66	D-67	D-68
Phosphorodithioic acid, *S*-(((p-chlorophenyl)thio)methyl) *O*,*O*-diethyl ester	D-38	D-43	D-44
Phosphorodithioic acid, *O*,*O*-dimethyl ester, *S*-ester with 3-(mercaptomethyl)-1,2,3-benzotriazin-4(3*H*)-one	D-108	D-117	D-118
Phosphorodithioic acid, *O*,*O*-dimethyl ester, *S*-ester with *N*-(mercaptomethyl)phthalimide	D-38	D-45	D-46
Phosphorodithioic acid, S,S'-methylene *O*,*O*,*O*',*O*'-tetraethyl ester	D-108	D-115	D-116
Phosphorothioic acid, *O*,*O*-diethyl *O*-(*p*-(methylsulfinyl) phenyl) ester	D-66	D-73	D-74
Phosphorothioic acid, *O*,*O*-dimethyl-, *O*-(4-(methylthio)-*m*-tolyl) ester	D-66	D-71	D-72
Propionitrile, 2-hydroxy-	D-75	D-76	D-77
Pyridine, 3-chloromethyl-, hydrochloride	D-80	D-81	D-82

(continued)

APPENDIX D
(continued)

CHROMATOGRAMS, MASS SPECTRA, AND CALIBRATION CURVES FOR GC/MS

Index

Compound	Reference chromatogram Figure No.	Reference mass spectrum Figure No.	Reference calibration curve Figure No.
Pyridine, 2,6-diamino-3-(phenylazo)-, monohydrochloride	D-89	D-106	D-107
Styrene	D-89	D-90	D-91
Succinic acid, mercapto-, diethyl ester, *S*-ester with *O*,*O*-dimethyl phosphorodithioate	D-108	D-111	D-112
Tetraethylpyrophosphate	D-108	D-109	D-110
Toluene, 2,4-diamino-	D-89	D-98	D-99
o-Toluidine	D-89	D-92	D-93
o-Toluidine, 5-chloro-	D-119	D-120	D-121
p-Toluidine, *N*-(2-chloroethyl)-2,6-dinitro-*N*-propyl-*alpha*,*alpha*,*alpha*-trifluoro-	D-124	D-125	D-126
o-Toluidine, 4-(o-tolylazo)-	D-51	D-62	D-63
p-Toluidine, *alpha*,*alpha*,*alpha*-trifluoro-2,6-dinitro-*N*,*N*-dipropyl-	D-127	D-128	D-129
S-Triazine, 2,4-dichloro-6-(*o*-chloroanilino)-	D-13	D-16	D-17

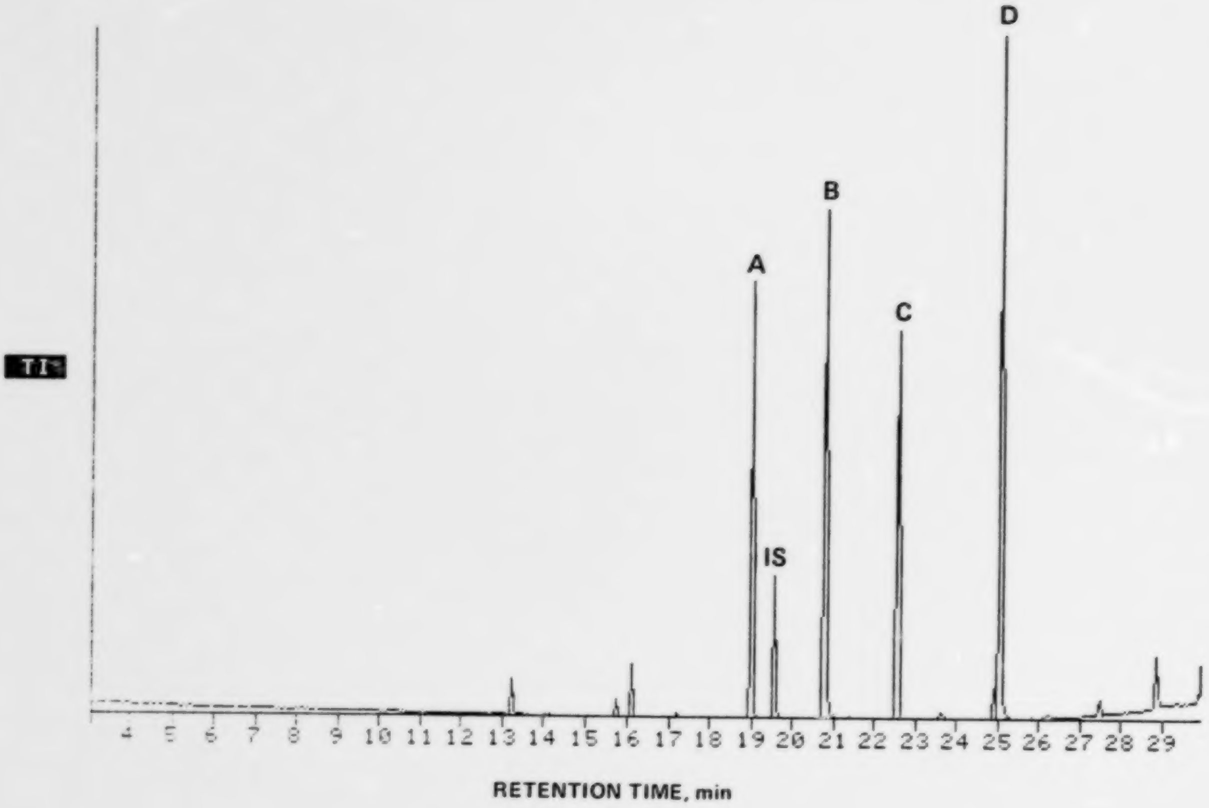

Figure D-1. Chromatogram by GC/MS for

(A) carbamic acid, methyl-, 2,3-dihydro-2,2-dimethyl-7-benzofuranyl ester(t_R = 19.0 min),
(B) carbamic acid, methyl-, 1-naphthyl ester(t_R = 20.8 min),
(C) 4-cyclohexene-1,2-dicarboximide, N-(trichloromethyl)thio-(t_R = 22.6 min), and
(D) aniline, 4,4'-methylenebis(N,N-dimethyl- (t_R = 25.1 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

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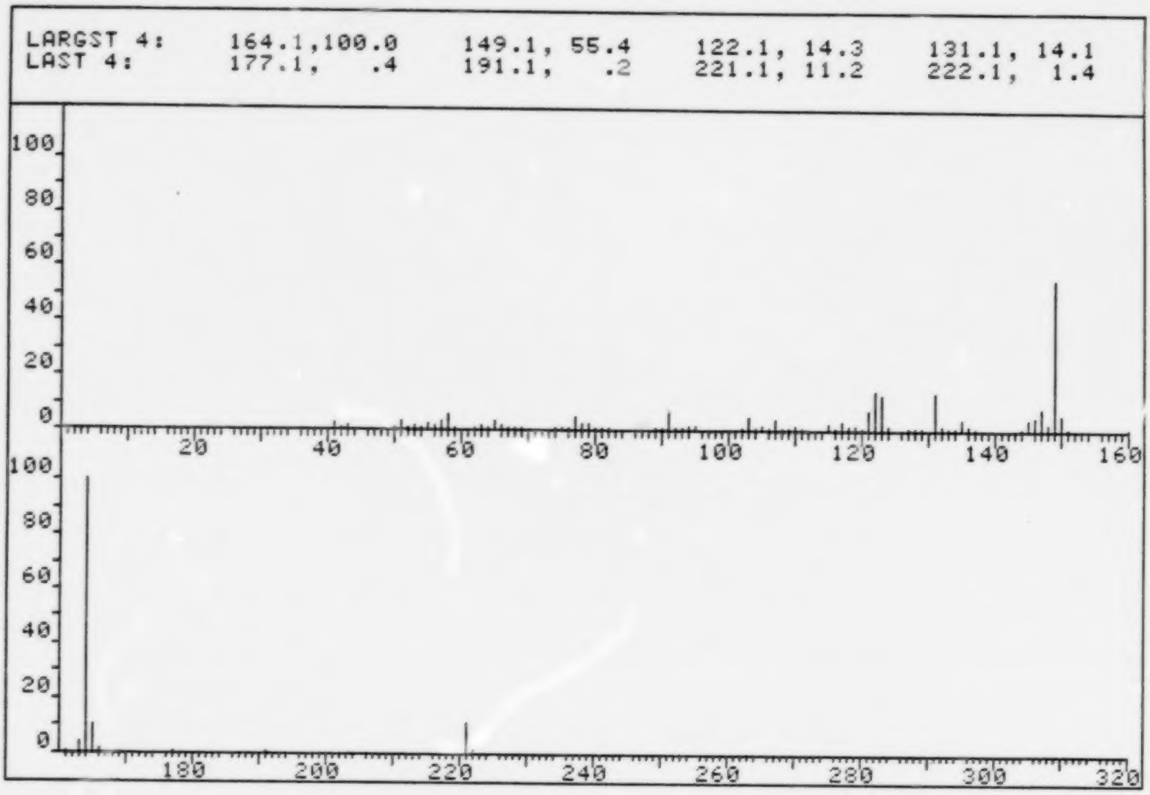

Figure D-2. Mass spectrum for carbamic acid, methyl-, 2,3-dihydro-2,2-dimethyl-7-benzofuranyl ester.

TABLE D-1. KEY IONS AND RELATIVE ABUNDANCES FOR CARBAMIC ACID, METHYL-, 2-3-DIHYDRO-2,2-DIMETHYL-7-BENZOFURANYL ESTER

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	2.3	79	2.5	115	2.3	136	1.9
43	1.5	80	.6	116	.7	137	.5
50	1.0	81	.6	117	3.4	145	4.4
51	3.2	89	.8	118	1.9	146	5.2
52	1.1	91	6.9	119	1.6	147	7.7
53	1.5	92	1.0	120	.6	148	2.6
55	2.4	93	1.2	121	7.2	149	55.4
56	1.6	94	2.0	122	14.3	150	5.5
57	3.0	95	1.5	123	12.8	151	.5
58	5.5	102	.9	124	1.8	163	4.2
63	1.5	103	5.3	127	.9	164	100.0
64	.8	104	.8	128	.6	165	10.1
65	2.9	105	1.9	131	14.1	166	.9
66	1.5	106	.8	132	1.8	221	11.2
67	.9	107	4.4	133	.5	222	1.4
77	5.1	108	1.1	134	.6		
78	2.2	110	1.7	135	3.9		

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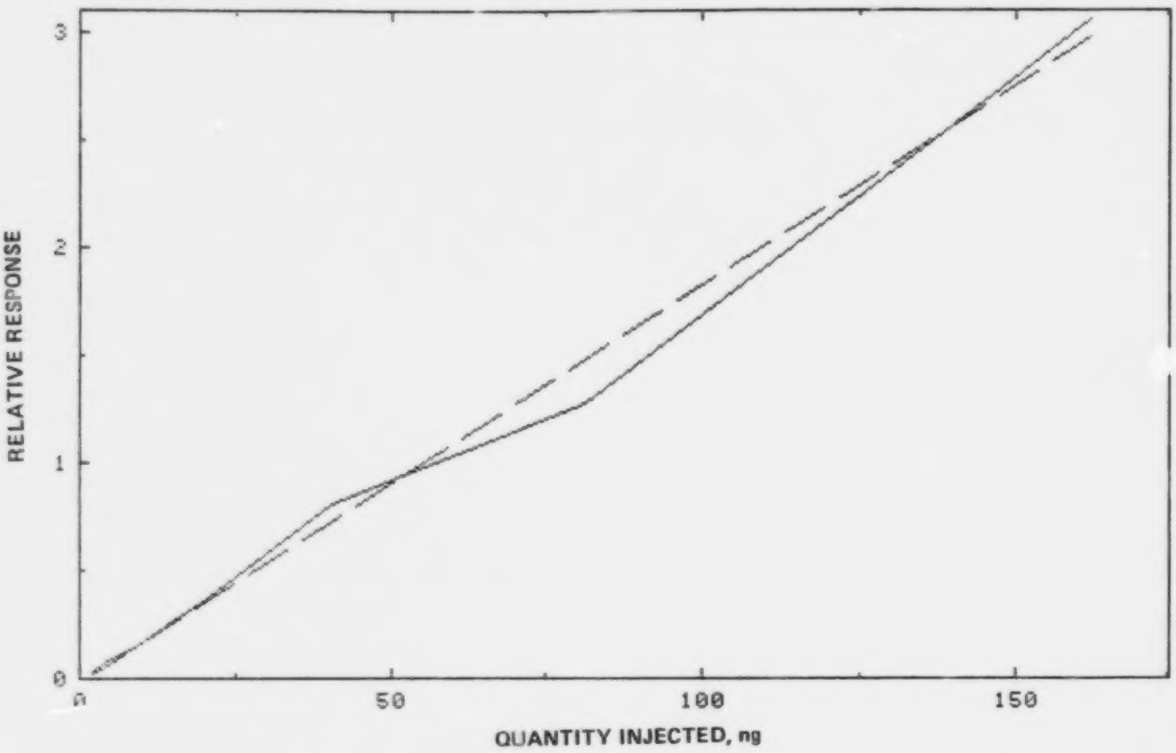
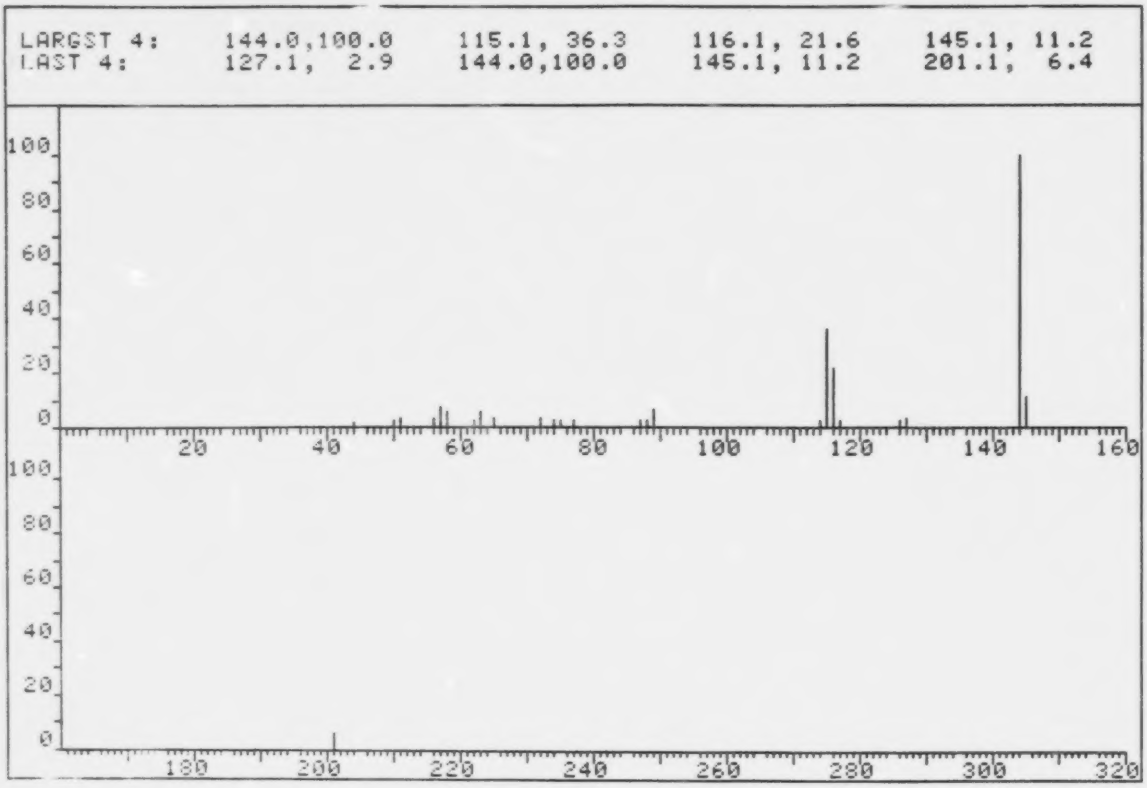


Figure D-3. Calibration curve for the determination of carbamic acid, methyl-, 2,3-dihydro-2,2-dimethyl-7-benzofuranyl ester by GC/MS.

Range: 8.1 - 160 ng
Correlation coefficient: 0.996
Slope: 1.8×10^{-2}
y-Intercept: -2.0×10^{-2}



```
LARGST 4:     144.0,100.0     115.1, 36.3     116.1, 21.6     145.1, 11.2
LAST 4:       127.1,   2.9    144.0,100.0     145.1, 11.2     201.1,  6.4
```

Figure D-4. Mass spectrum for carbamic acid, methyl-, 1-naphthyl ester.

TABLE D-2. KEY IONS AND RELATIVE ABUNDANCES FOR CARBAMIC ACID, METHYL-, 1-NAPHTHYL ESTER

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	2.0	63	5.5	88	2.2	127	2.9
50	2.2	65	3.5	89	6.8	144	100.0
51	3.1	72	2.9	114	2.2	145	11.2
56	3.3	74	2.6	115	36.3	201	6.4
57	7.3	75	2.2	116	21.6		
58	5.5	77	2.4	117	2.4		
62	2.4	87	2.2	126	2.2		

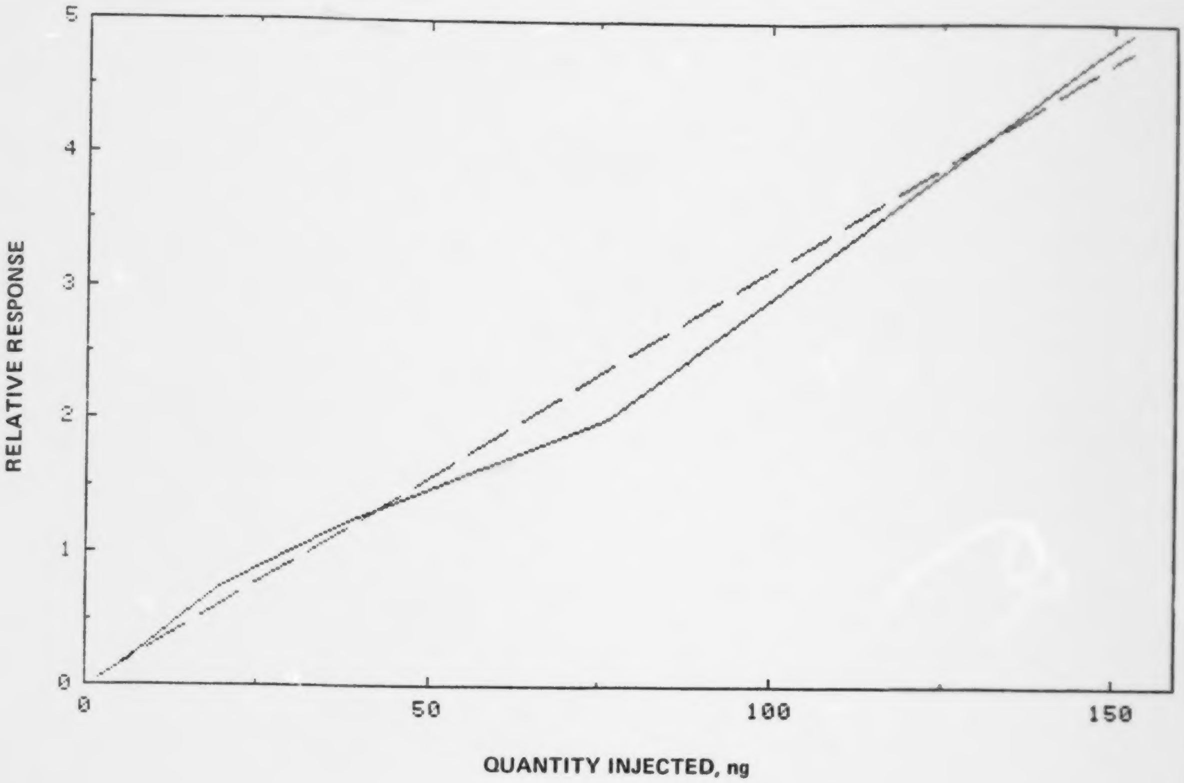


Figure D-5. Calibration curve for the determination of carbamic acid, methyl-, 1-naphthyl ester by GC/MS.

Range: 1.9 - 150 ng
Correlation coefficient: 0.995
Slope: 3.1×10^{-2}
y-Intercept: -1.7×10^{-2}

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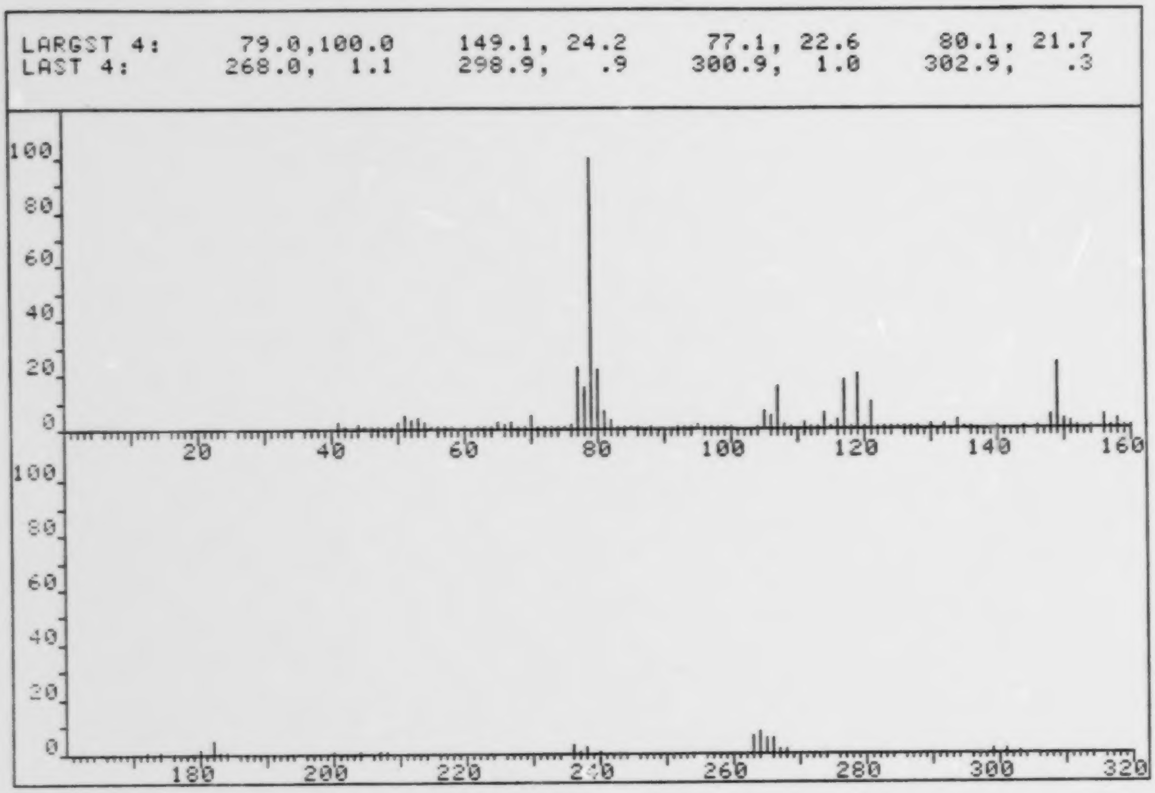


```
LARGST 4:     79.0,100.0     149.1, 24.2      77.1, 22.6      80.1, 21.7
LAST 4:      268.0,  1.1     298.9,   .9     300.9,  1.0     302.9,   .3
```

Figure D-6. Mass spectrum for 4-cyclohexene-1,2-dicarboximide, N-(trichloromethyl)thio- .

TABLE D-3. KEY IONS AND RELATIVE ABUNDANCES FOR 4-CYCLOHEXENE-1,2-DICARBOXIMIDE, *N*-(TRICHLOROMETHYL)THIO-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	2.1	71	.5	107	15.5	151	2.7
42	.7	72	1.3	108	1.8	154	.9
44	2.0	76	2.0	109	.7	156	5.1
45	.5	77	22.6	111	2.1	158	3.2
46	1.0	78	15.1	114	5.6	160	1.0
47	1.2	79	100.0	115	.7	172	.6
49	.6	80	21.7	116	3.5	180	1.0
50	2.2	81	6.4	117	17.9	182	4.3
51	4.8	82	2.9	118	1.0	184	.5
52	3.4	83	.6	119	20.0	236	2.7
53	4.1	84	1.2	121	9.6	238	1.8
54	2.3	86	.8	122	1.0	263	6.4
55	1.1	93	1.0	123	1.2	264	8.3
56	.7	94	.7	125	.7	265	5.1
60	1.0	95	1.8	130	1.8	266	5.7
63	.9	97	.8	132	1.4	267	1.4
65	2.6	98	.6	134	3.3	268	1.1
66	1.3	99	.6	136	1.0	299	.9
67	2.2	104	.8	148	4.8	301	1.0
69	.5	105	6.8	149	24.2		
70	5.3	106	5.3	150	3.0		

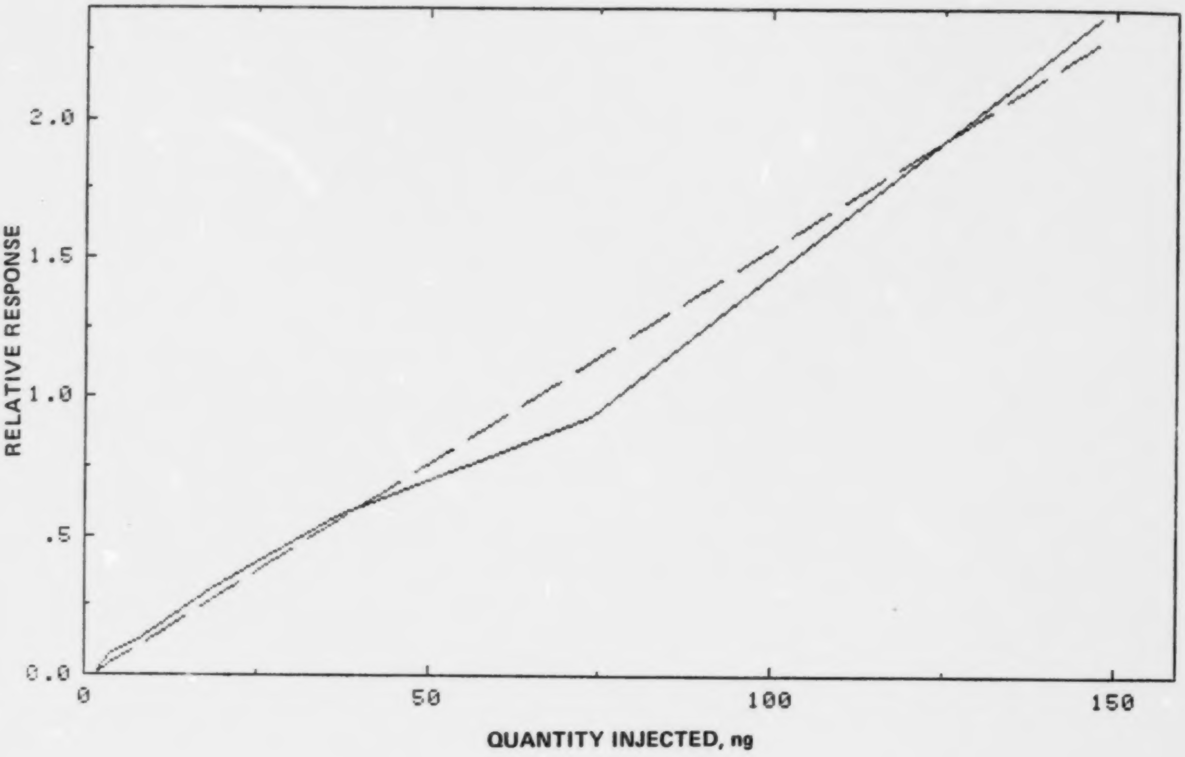
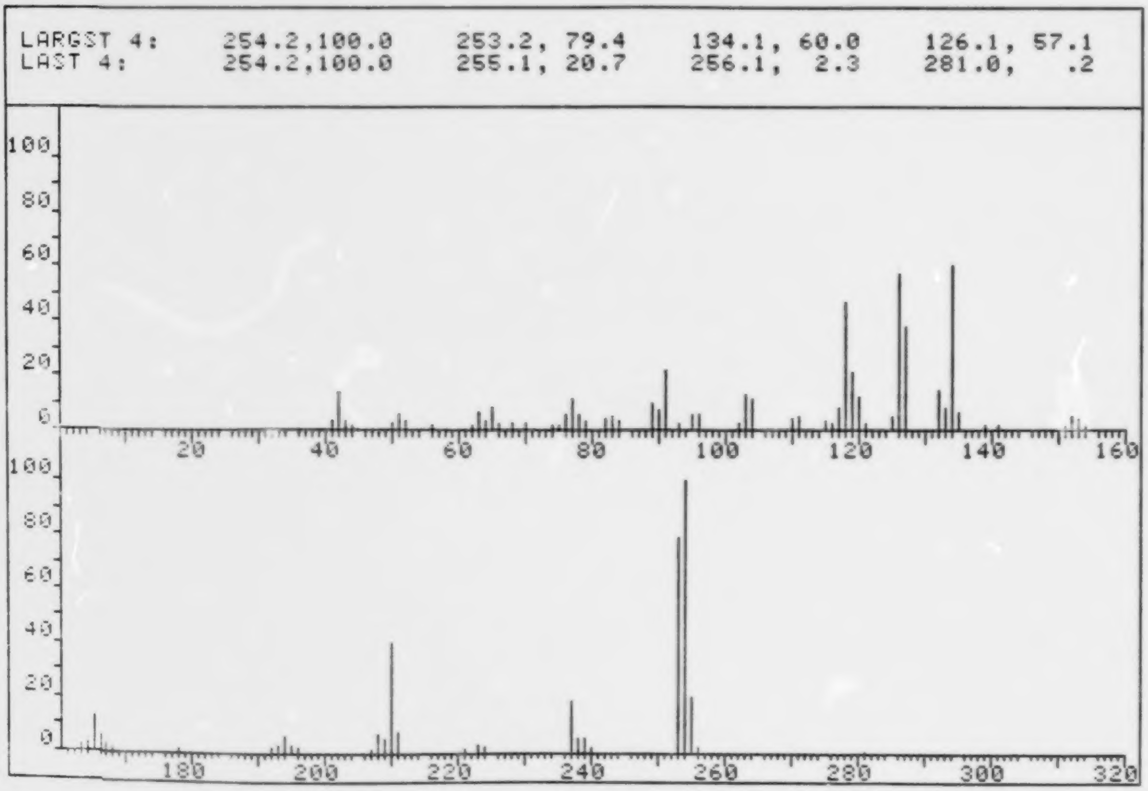


Figure D-7. Calibration curve for the determination of 4-cyclohexene-1,2-dicarboximide, N-(trichloromethyl)thio- by GC/MS.

Range: 1.9 - 150 ng
Correlation coefficient: 0.993
Slope: 1.6×10^{-2}
y-Intercept: -3.4×10^{-2}



```
LARGST 4:     254.2,100.0     253.2, 79.4     134.1, 60.0     126.1, 57.1
LAST 4:       254.2,100.0     255.1, 20.7     256.1,  2.3     281.0,   .2
```

Figure D-8. Mass spectrum for aniline, 4,4'-methylene-bis(*N*,*N*-dimethyl-.

TABLE D-4. KEY IONS AND RELATIVE ABUNDANCES FOR ANILINE, 4-4'-METHYLENEBIS-(*N*,*N*-DIMETHYL-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	3.3	82	4.6	125	4.9	193	2.8
42	13.8	83	5.2	126	57.1	194	6.0
43	3.3	84	3.3	127	38.0	195	3.3
44	1.8	89	9.6	132	14.5	196	1.8
50	2.8	90	7.5	133	8.0	207	1.6
51	6.0	91	21.3	134	60.0	208	6.5
52	3.3	93	2.3	135	6.8	209	5.7
56	1.8	95	5.9	139	1.9	210	40.8
62	1.8	96	5.9	141	1.6	211	7.5
63	6.7	102	2.8	151	1.9	221	1.6
64	2.9	103	12.5	152	4.9	223	3.3
65	7.8	104	11.5	153	3.9	224	2.1
66	2.1	110	4.1	154	1.8	237	18.9
68	2.1	111	4.9	163	1.9	238	5.5
70	2.1	115	3.7	164	2.6	239	5.2
74	1.8	116	2.6	165	13.0	240	2.1
75	1.9	117	8.1	166	5.7	253	79.3
76	5.5	118	46.7	167	2.6	254	100.0
77	11.2	119	20.6	168	1.6	255	20.6
78	5.5	120	12.0	178	1.6	256	2.3
79	3.3	121	2.1	192	1.8		

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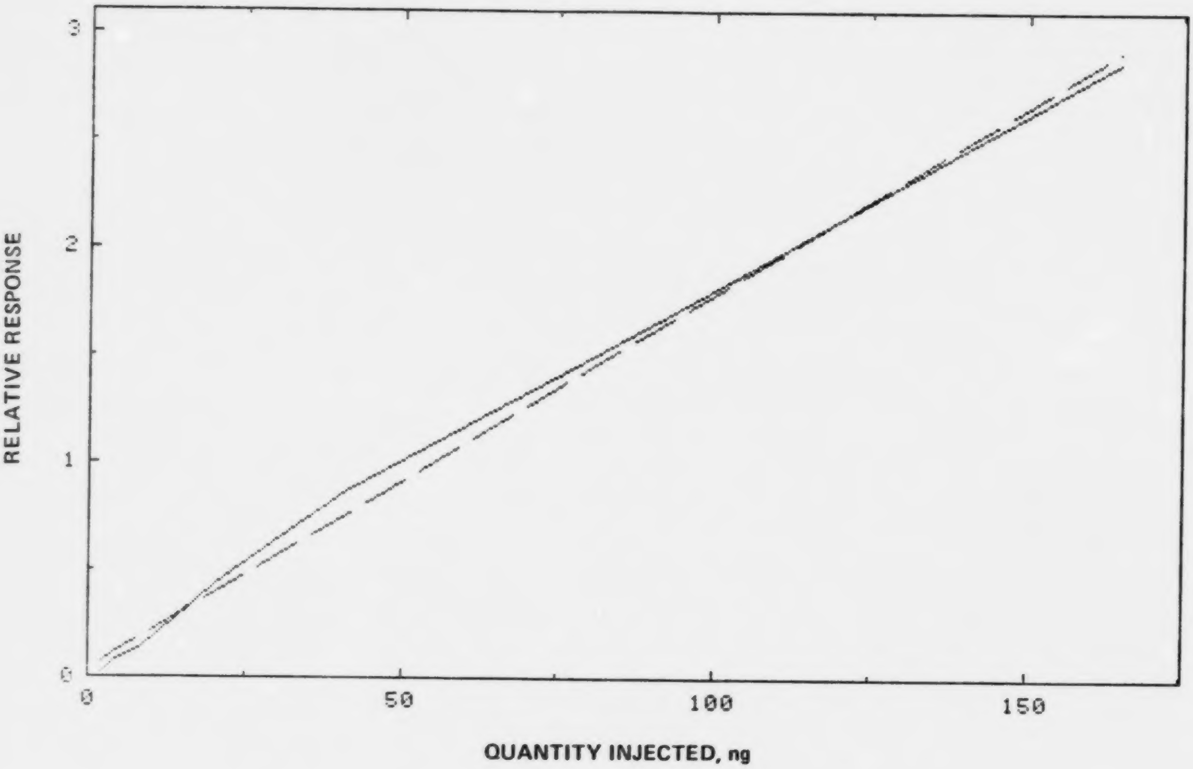


Figure D-9. Calibration curve for the determination of aniline, 4,4'-methylenebis(N,N-dimethyl- by GC/MS.

Range: 8.2 - 160 ng
Correlation coefficient: 0.999
Slope: 1.7×10^{-2}
y-Intercept: 3.2×10^{-2}

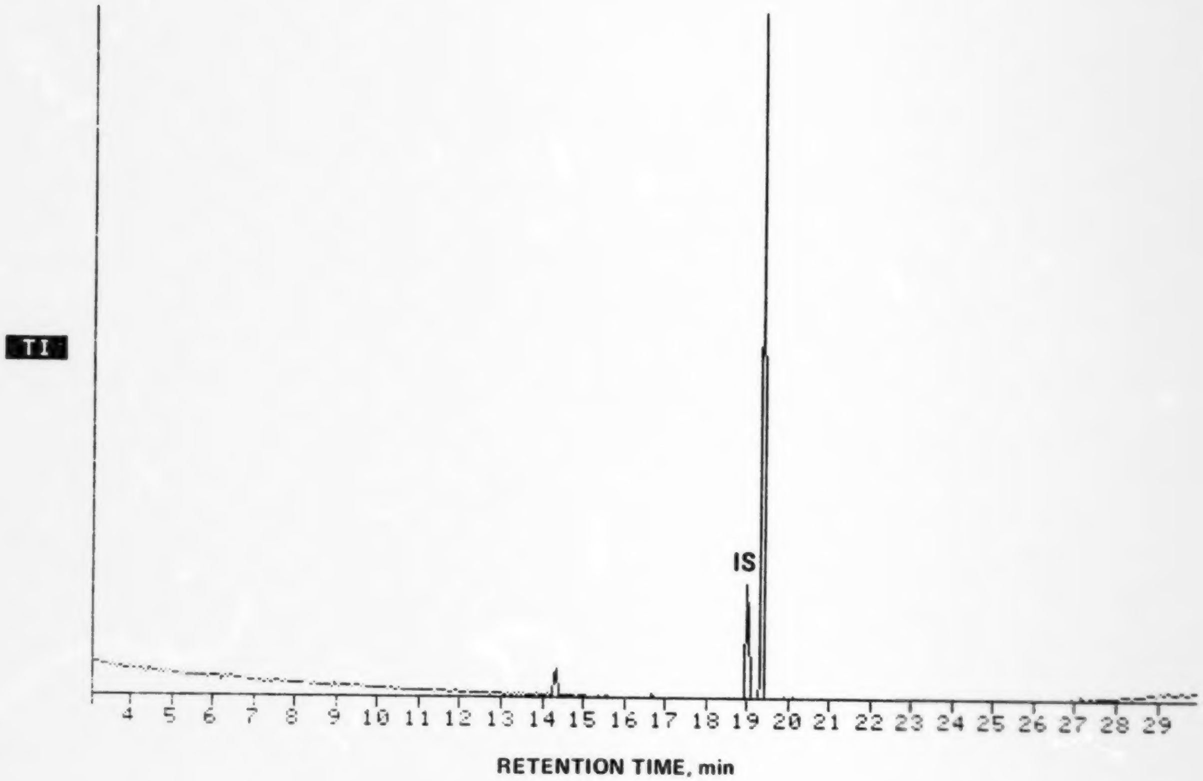
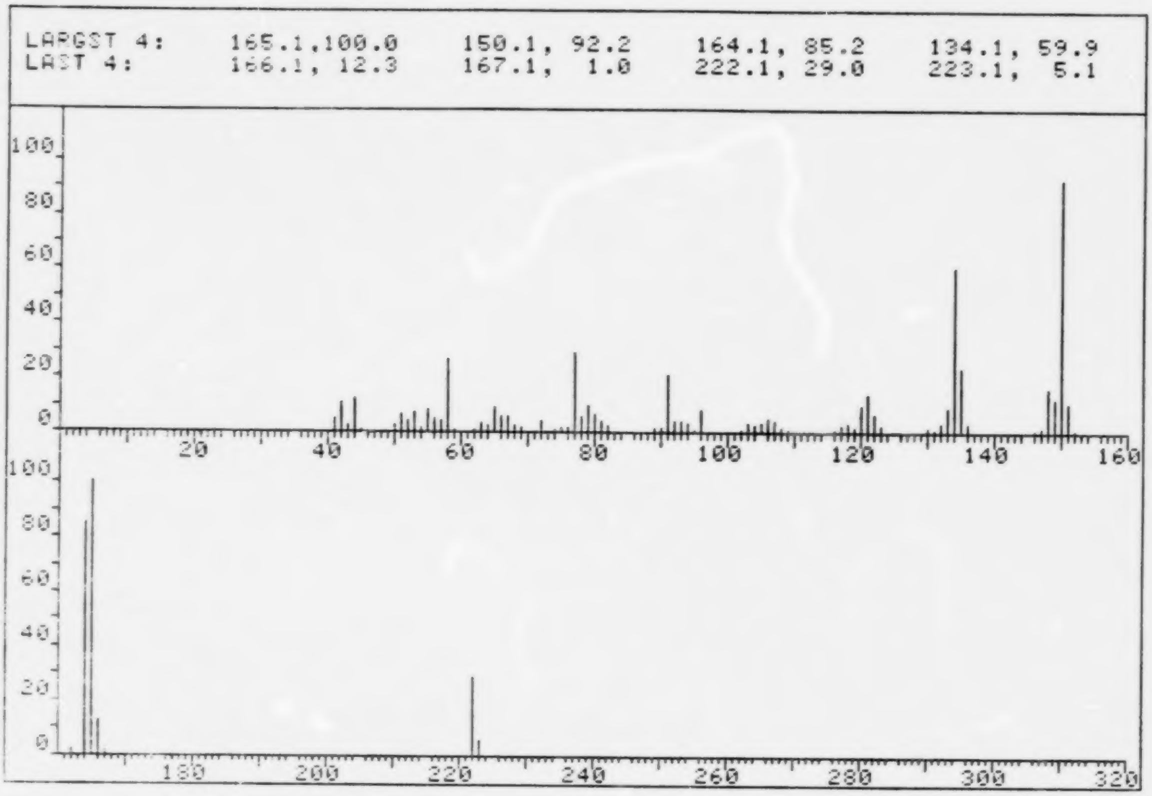


Figure D-10. Chromatogram by GC/MS for carbamic acid, methyl-, 4-dimethylamino-3,5-xylyl ester(t_R = 19.4 min).

Range: 1.8 - 140 ng
Correlation coefficient: 0.999
Slope: 1.4×10^{-2}
y-Intercept: -1.1×10^{-2}

TABLE D-5. KEY IONS AND RELATIVE ABUNDANCES FOR CARBAMIC ACID, METHYL-, 4-DIMETHYLAMINO-3,5-XYLYL

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	5.3	67	5.5	95	1.2	132	3.8
42	10.6	68	2.1	96	8.1	133	9.2
43	2.6	69	1.5	102	1.0	134	59.9
44	12.3	72	4.3	103	3.0	135	23.3
45	.9	74	.9	104	2.2	136	3.8
50	2.1	74	1.2	105	3.0	146	1.1
51	6.4	75	1.3	106	4.7	147	1.7
52	4.0	76	1.5	107	4.2	148	16.2
53	6.9	77	29.3	108	1.8	149	12.0
54	1.8	78	5.8	109	.9	150	92.2
55	8.6	79	9.5	116	.9	151	10.7
56	4.9	80	6.8	117	2.4	152	1.1
57	4.3	81	4.0	118	3.6	162	1.8
58	26.7	82	2.1	119	1.9	164	85.2
59	1.2	89	2.0	120	10.0	165	100.0
62	1.6	90	1.4	121	13.7	166	12.3
63	3.4	91	21.2	122	6.6	167	1.0
64	2.2	92	3.9	123	2.4	222	29.0
65	9.1	93	4.0	130	1.7	223	5.1
66	5.8	94	3.1	131	1.0		



```
LARGST 4:     165.1,100.0     150.1, 92.2     164.1, 85.2     134.1, 59.9
LAST 4:       166.1, 12.3     167.1,  1.0     222.1, 29.0     223.1,  5.1
```

Figure D-11. Mass spectrum for carbamic acid, methyl-, 4-dimethylamino-3,5-xylyl ester.

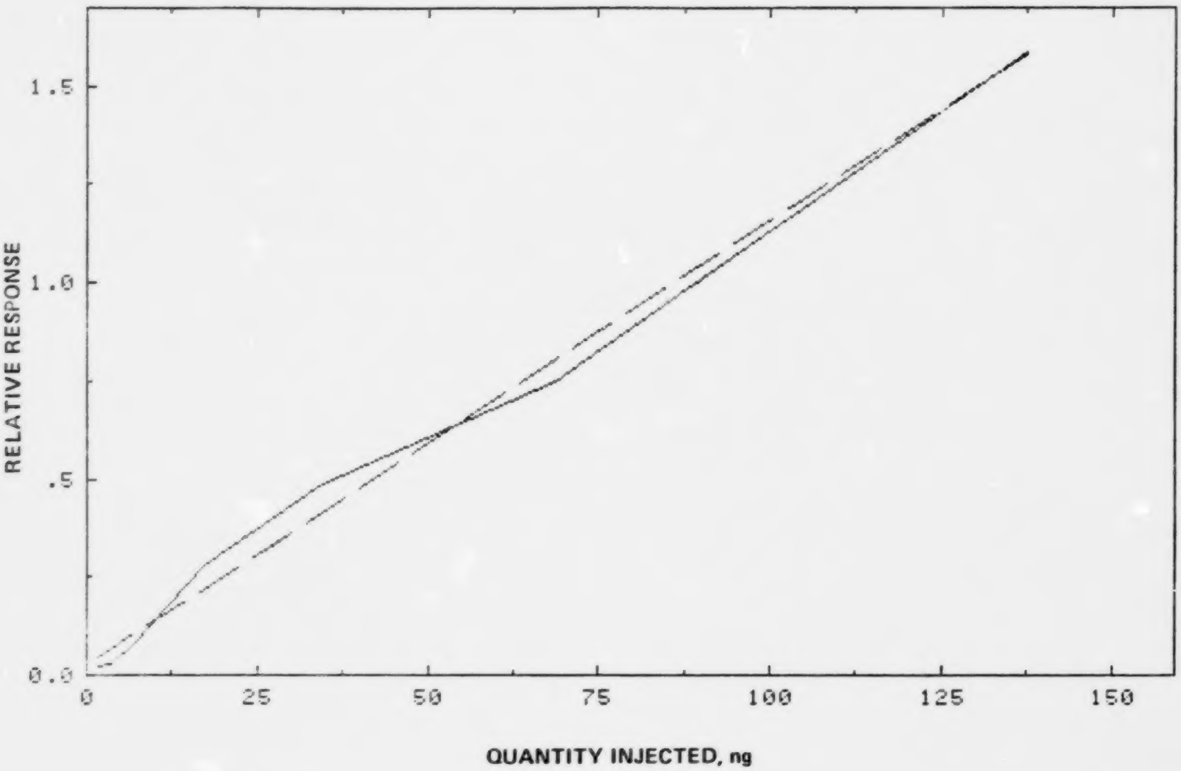


Figure D-12. Calibration curve for the determination of carbamic acid, methyl-, 4-dimethylamino-3,5-xylyl ester by GC/MS.

Range: 6.9 - 140 ng
Correlation coefficient: 0.996
Slope: 1.1×10^{-2}
y-Intercept: 2.6×10^{-2}

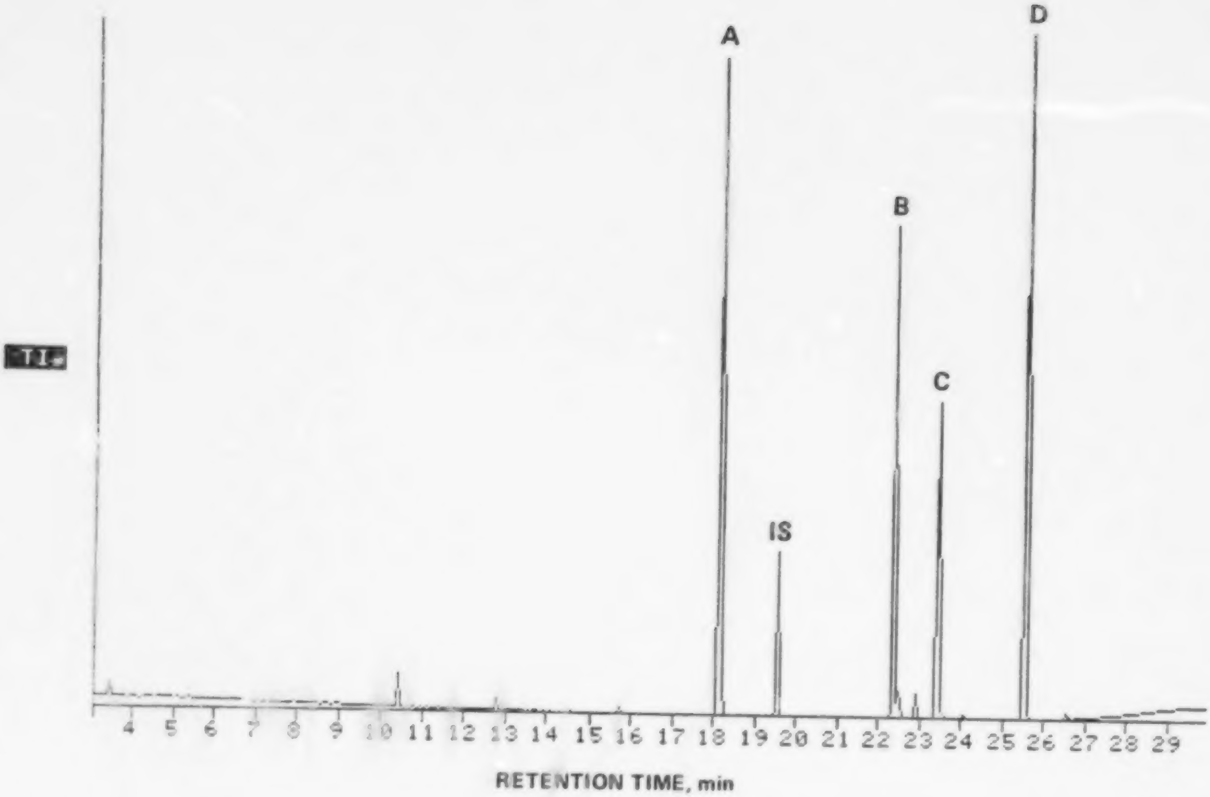
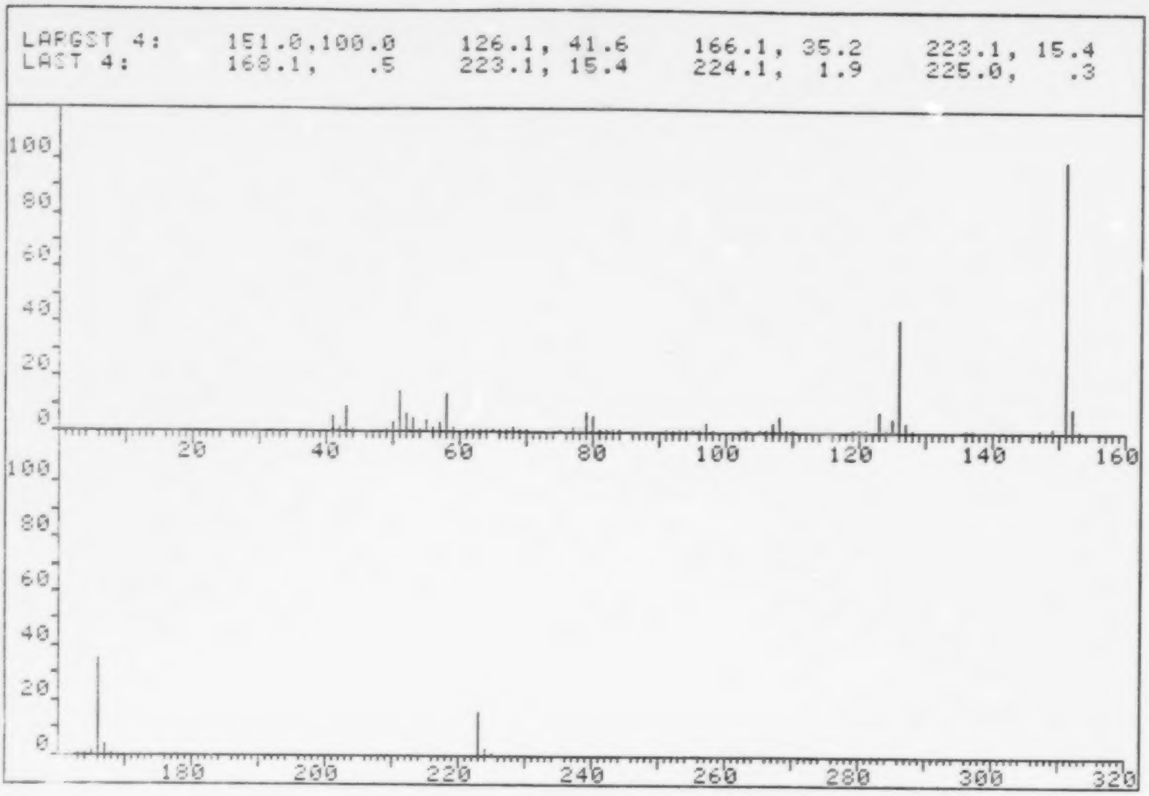


Figure D-13. Chromatogram by GC/MS for

(A) carbamic acid, methyl-, 2,3-(dimethylmethylenedioxy)-phenyl ester(t_R = 18.2 min),
(B) *S*-triazine, 2,4-dichloro-6-(*o*-chloroanilino)-(t_R = 22.4 min),
(C) carbanilic acid, *m*-chloro-, 4-chloro-2-butynyl ester(t_R = 23.4 min), and
(D) 4-cyclohexene-1,2-dicarboximide, *N*-((1,1,2,2-tetrachloroethyl)thio)- (t_R = 25.6 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal



```
LARGST 4:     151.0,100.0     126.1, 41.6     166.1, 35.2     223.1, 15.4
LAST 4:       168.1,    .5    223.1, 15.4     224.1,  1.9     225.0,    .3
```

Figure D-14. Mass spectrum for carbamic acid, methyl-, 2,3-(dimethylmethylenedioxy)phenyl ester.

TABLE D-6. KEY IONS AND RELATIVE ABUNDANCES FOR CARBAMIC ACID, METHYL-, 2,3-(DIMETHYLMETHYLENEDIOXY) PHENYL ESTER

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	5.7	62	.7	95	1.1	137	.5
42	1.4	63	.9	96	.8	149	1.8
43	9.0	65	.5	97	3.3	151	100.0
50	3.3	66	.5	107	3.1	152	9.1
51	14.8	67	.7	108	5.4	153	.9
52	6.2	68	1.7	109	1.2	164	.5
53	5.0	77	1.5	121	.7	165	1.5
54	.8	78	.7	123	7.1	166	35.2
55	4.3	79	7.5	124	1.0	167	3.6
56	1.8	80	5.5	125	4.9	168	.5
57	3.6	81	1.3	126	41.6	223	15.4
58	14.1	91	.7	127	3.4	224	1.9
59	1.5	93	.7	128	.5		

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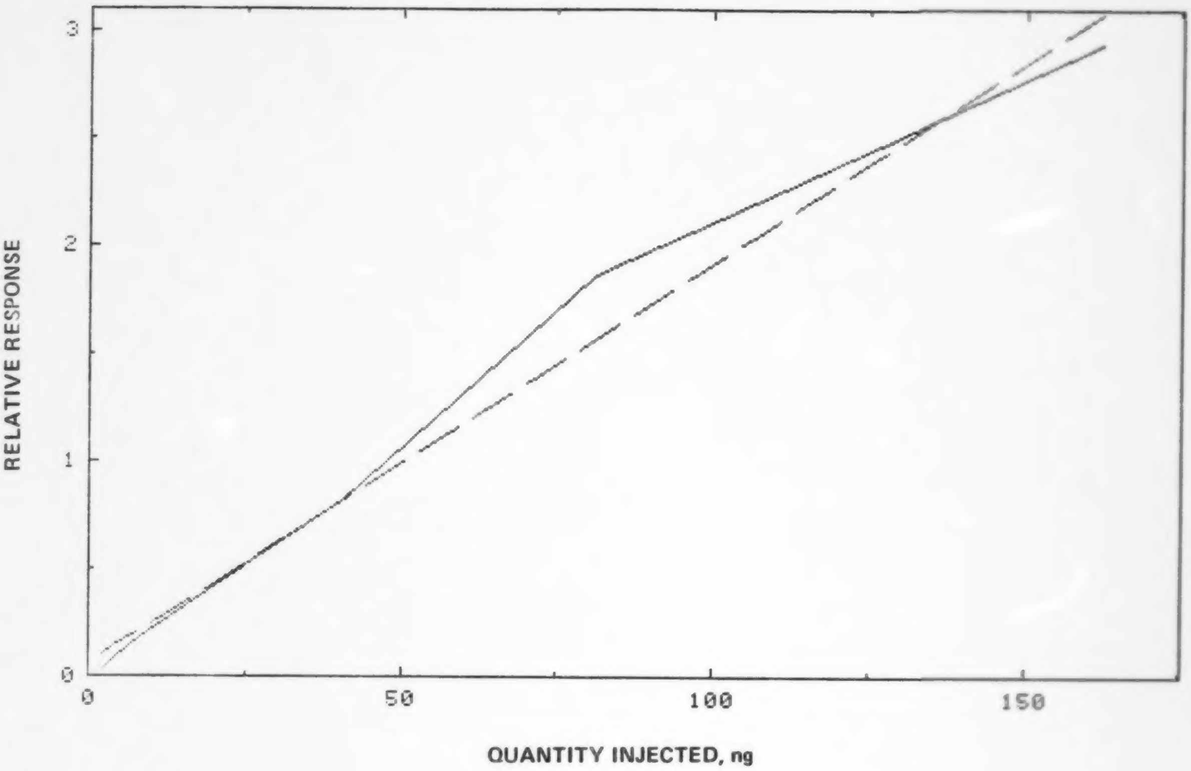


Figure D-15. Calibration curve for the determination of carbamic acid, methyl-, 2,3-(dimethylmethylenedioxy)phenyl ester by GC/MS.

Range: 8.1 - 160 ng
Correlation coefficient: 0.992
Slope: 1.8×10^{-2}
y-Intercept: 6.3×10^{-2}

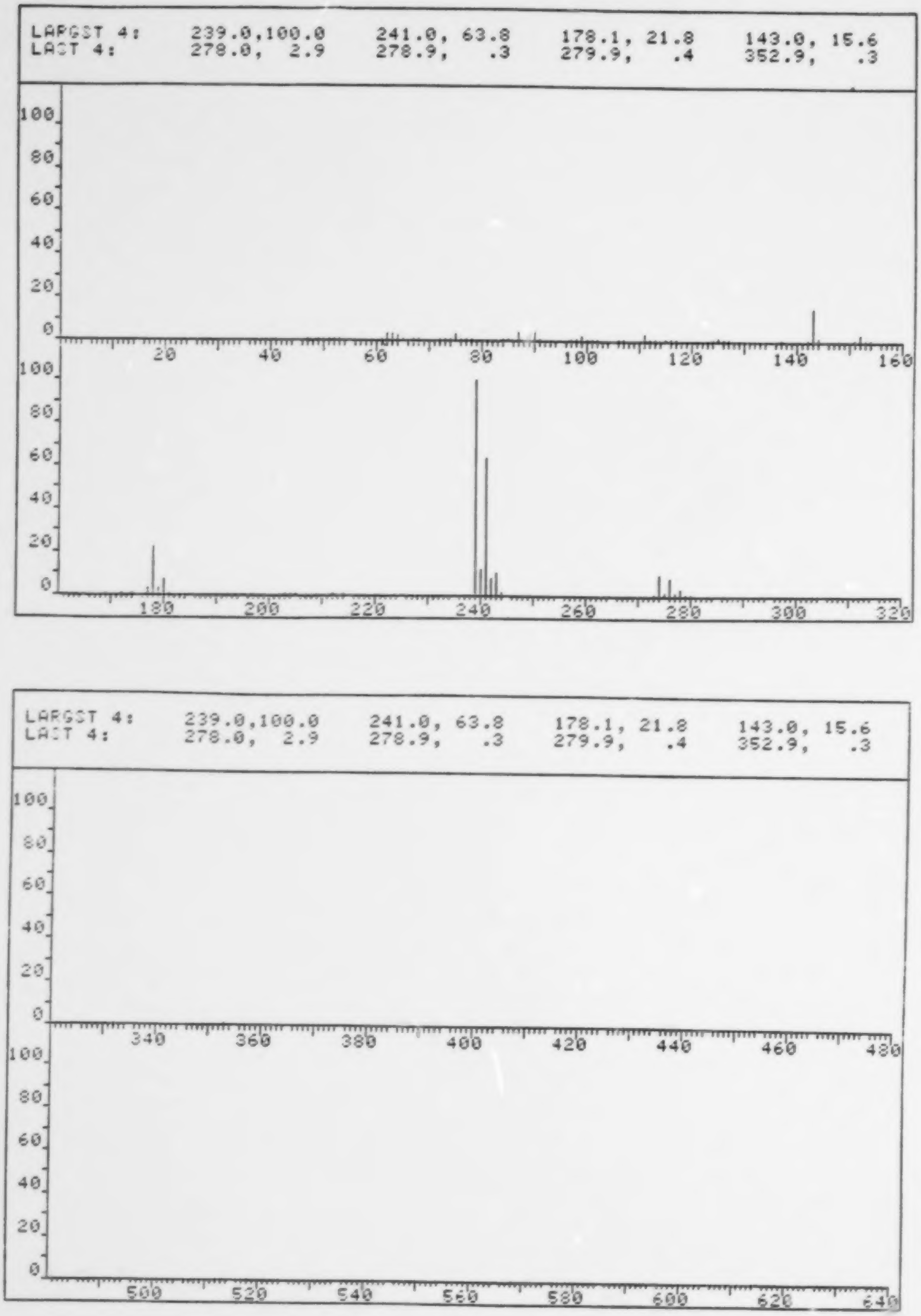


Figure D-16. Mass spectrum for S-triazine, 2,4-dichloro-6-(o-chloroanilino)-.

TABLE D-7. KEY IONS AND RELATIVE ABUNDANCES FOR S-TRIAZINE, 2,4-DICHLORO-6-(o-CHLOROANILINO)-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
50	1.3	89	3.4	143	15.6	214	.6
51	.7	90	4.5	144	1.7	239	100.0
52	.9	91	.9	151	1.1	240	11.8
53	.5	99	2.4	152	2.8	241	63.8
61	.9	101	.8	153	.7	242	7.3
62	3.2	106	.9	154	1.0	243	10.0
63	2.8	111	2.8	169	.7	244	1.2
64	2.1	113	.8	172	.8	273	.6
65	.8	115	.5	174	.5	274	9.4
73	1.2	116	.8	177	2.9	275	1.6
74	1.1	117	1.2	178	21.8	276	8.1
75	3.5	124	.9	179	2.7	277	1.0
76	1.0	125	1.3	180	6.9	278	2.9
77	.5	126	.5	181	.7		
85	.5	127	.7	203	.9		
87	4.0	142	.8	212	.7		

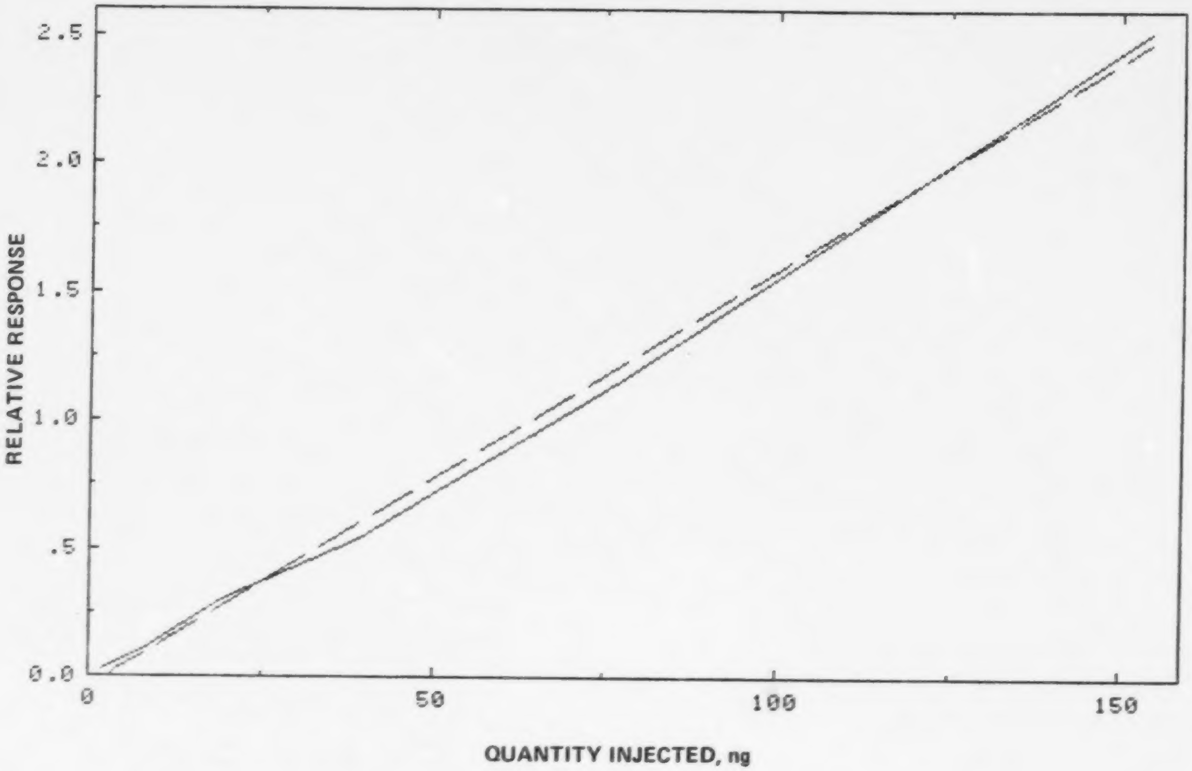
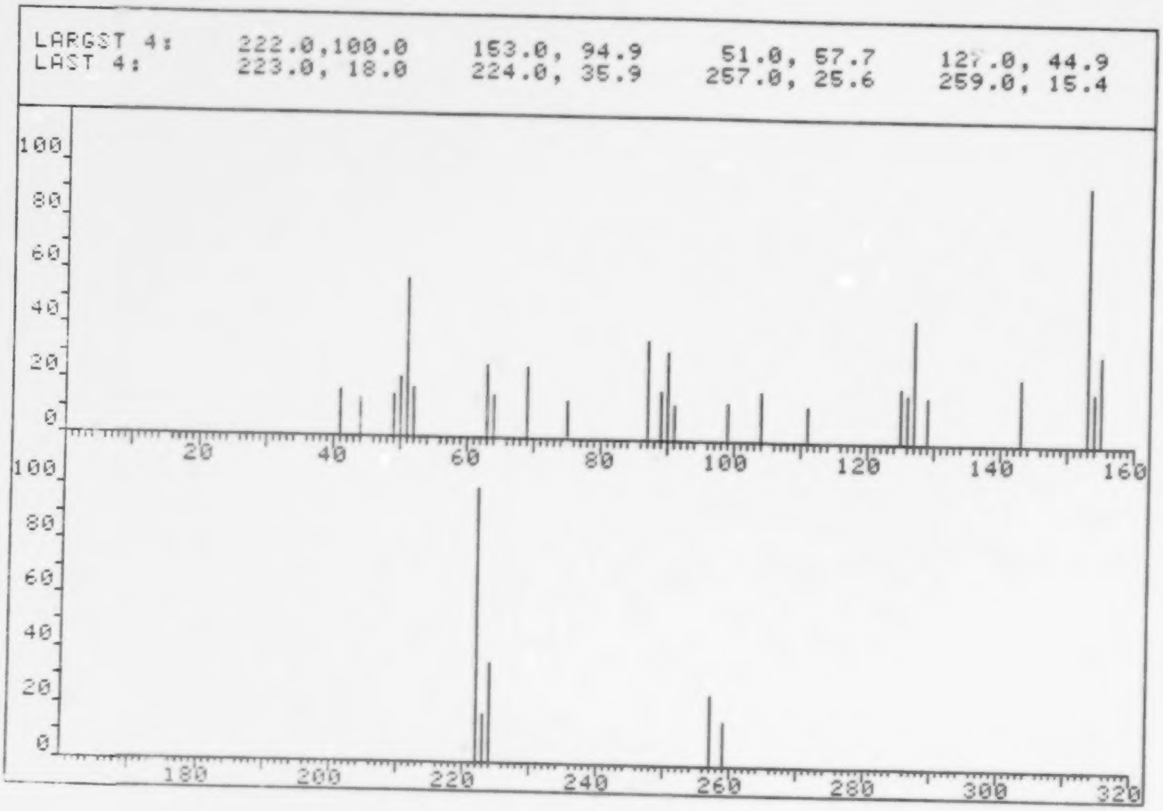


Figure D-17. Calibration curve for the determination of S-triazine, 2,4-dichloro-6-(o-chloroanilino)- by GC/MS.

Range: 7.7 - 140 ng
Correlation coefficient: 0.999
Slope: 1.6×10^{-2}
y-Intercept: -3.7×10^{-2}



```
LARGST 4:     222.0,100.0      153.0, 94.9       51.0, 57.7      127.0, 44.9
LAST 4:       223.0, 18.0      224.0, 35.9      257.0, 25.6      259.0, 15.4
```

Figure D-18. Mass spectrum for carbanilic acid, *m*-chloro-, 4-chloro-2-butynyl ester.

TABLE D-8. KEY IONS AND RELATIVE ABUNDANCES FOR CARBANILIC ACID, *m*-CHLORO-, 4-CHLORO-2-BUTYNYL ESTER

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	16.7	69	25.6	111	12.8	155	33.3
44	14.1	75	14.1	125	20.5	222	100.0
49	15.4	87	35.9	126	18.0	223	18.0
50	21.8	89	18.0	127	44.9	224	35.9
51	57.7	90	32.0	129	16.7	257	25.6
52	18.0	91	12.8	143	24.4	259	15.4
63	26.9	99	14.1	153	94.9		
64	15.4	104	18.0	154	19.2		

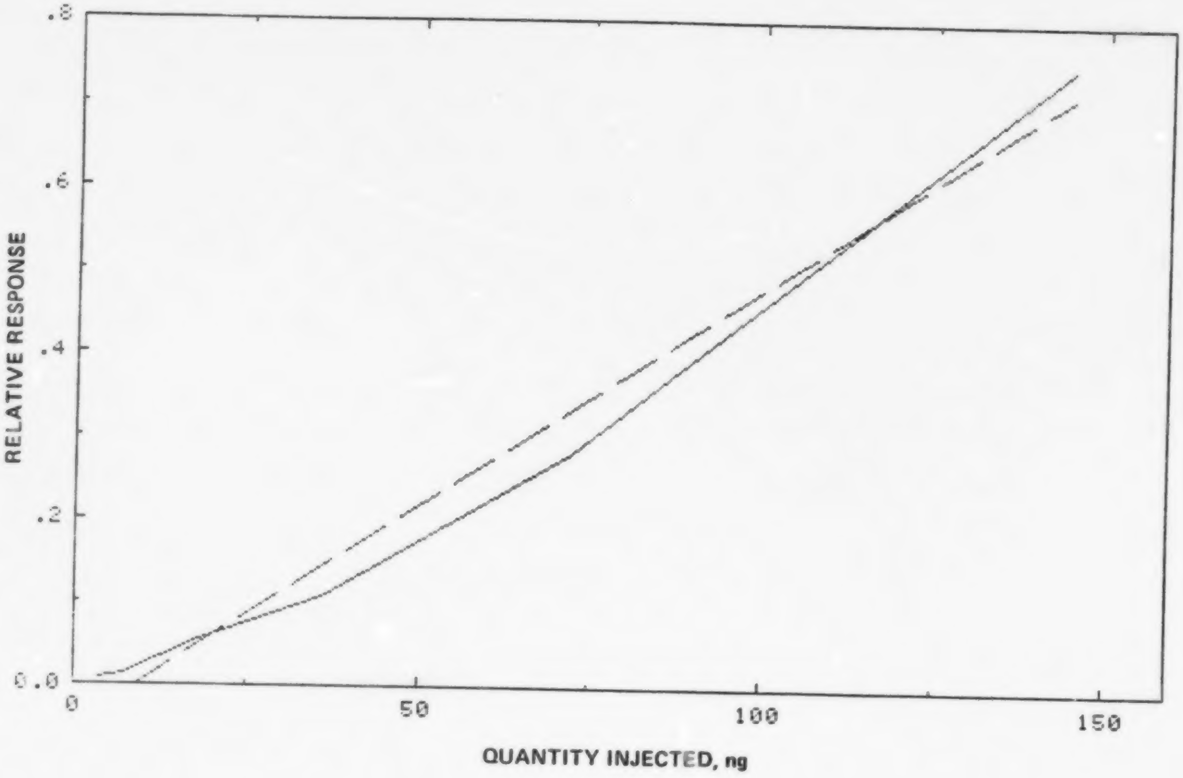


Figure D-19. Calibration curve for the determination of carbanilic acid, m-chloro-, 4-chloro-2-butynyl ester by GC/MS.

Range: 18 - 140 ng
Correlation coefficient: 0.989
Slope: 5.2×10^{-3}
y-Intercept: -3.8×10^{-2}

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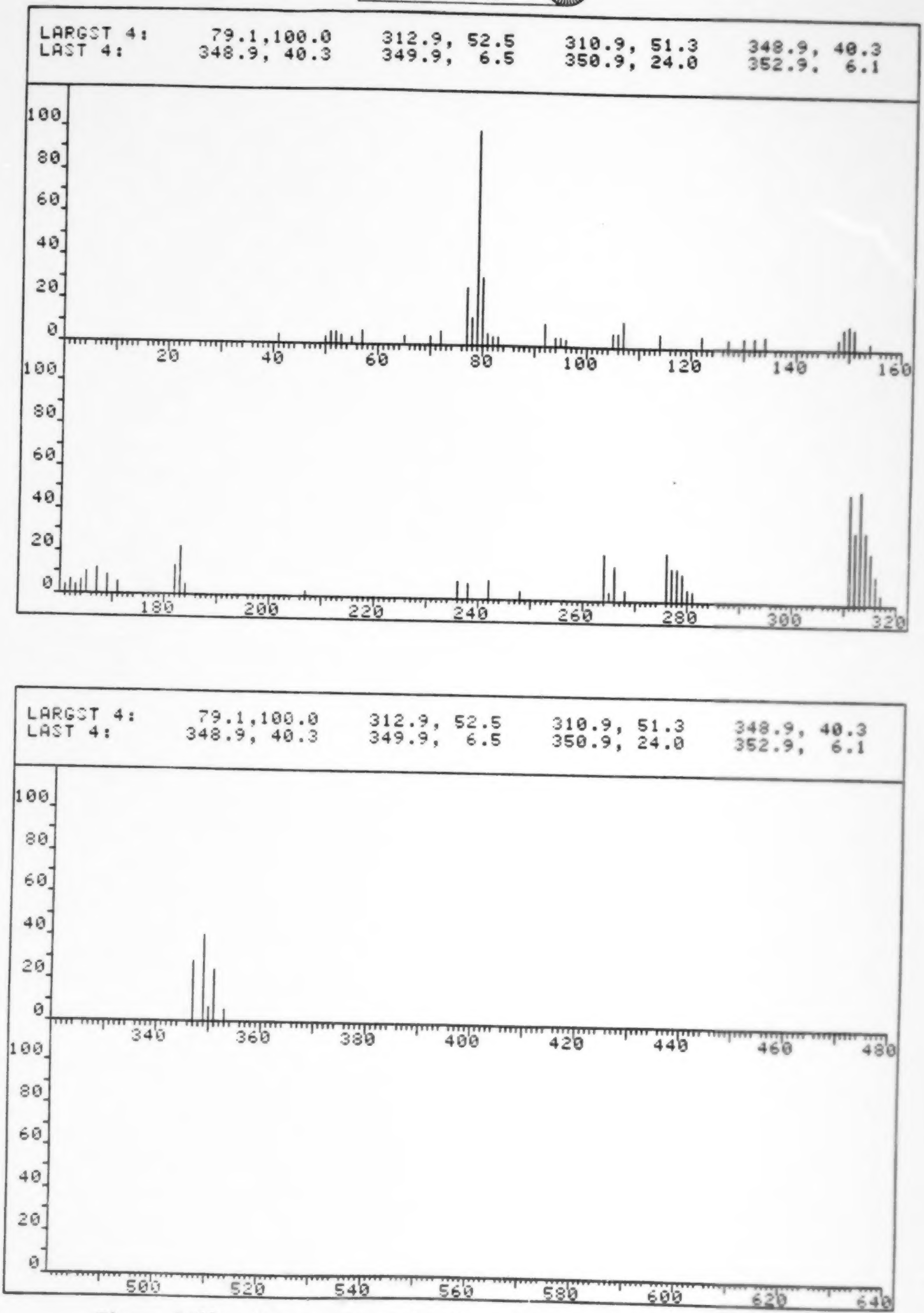


Figure D-20. Mass spectrum for 4-cyclohexene-1,2-dicarboximide, N-((1,1,2,2-tetrachloroethyl)thio)-.

TABLE D-9. KEY IONS AND RELATIVE ABUNDANCES FOR 4-CYCLOHEXENE-1,2-DICARBOXIMIDE, N-((1,1,2,2-TETRACHLOROETHYL)THIO)-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	4.2	95	4.6	164	6.1	278	15.2
50	3.8	96	3.8	165	9.9	279	12.5
51	5.7	105	6.8	167	11.8	280	5.7
52	5.7	106	6.5	169	8.7	281	4.6
53	4.6	107	12.2	171	5.3	311	51.3
55	3.8	114	6.8	182	13.7	312	33.8
57	6.8	122	5.7	183	22.1	313	52.5
65	4.2	127	4.6	184	4.6	314	33.5
70	4.2	130	5.3	207	1.9	315	23.6
72	6.8	132	4.9	236	8.0	316	13.3
77	27.0	134	5.7	238	6.8	317	4.6
78	12.5	148	5.3	242	8.4	347	28.5
79	100.0	149	9.9	248	3.8	349	40.3
80	31.2	150	11.0	264	21.3	350	6.5
81	5.7	151	9.9	265	3.8	351	24.0
82	4.6	154	3.8	266	15.6	353	6.1
83	4.2	161	4.2	268	4.6		
92	10.3	162	6.5	276	22.1		
94	4.2	163	4.2	277	14.8		

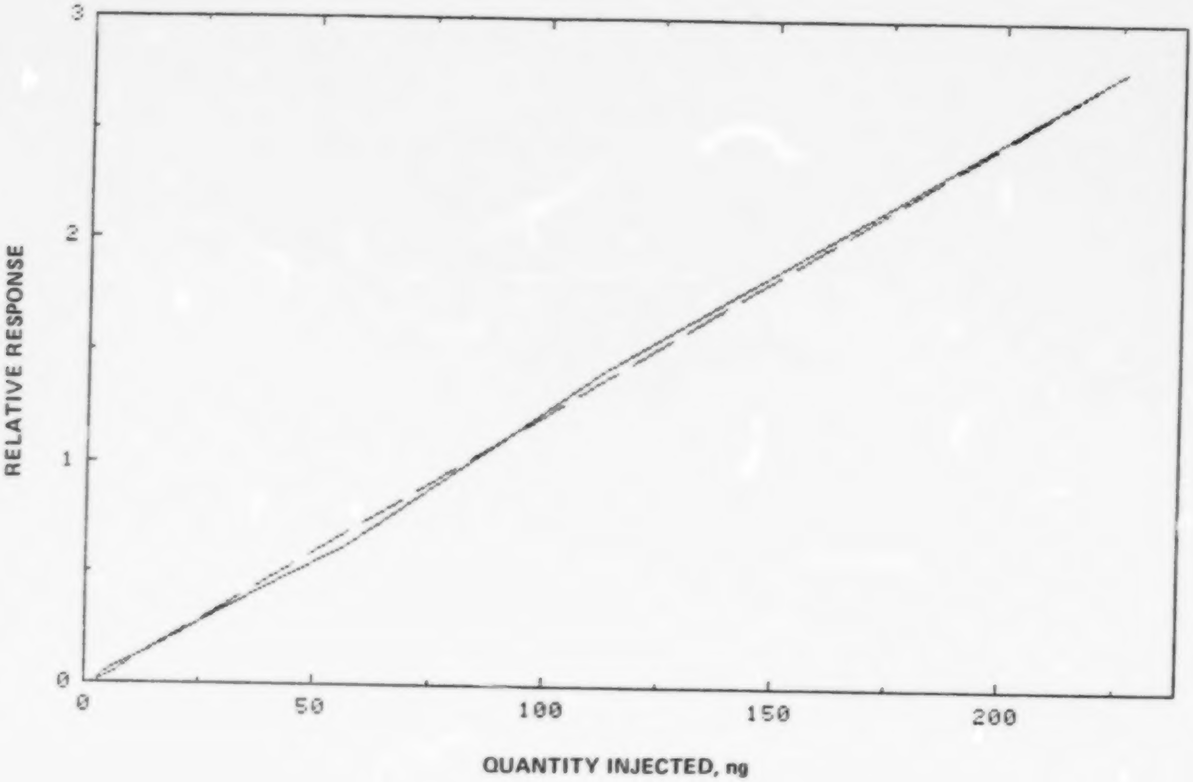


Figure D-21. Calibration curve for the determination of 4-cyclohexene-1,2-dicarboximide, N-((1,1,2,2-tetrachloroethyl)thio)- by GC/MS.

Range: 11 - 230 ng
Correlation coefficient: 0.999
Slope: 1.2×10^{-2}
y-Intercept: -2.6×10^{-2}

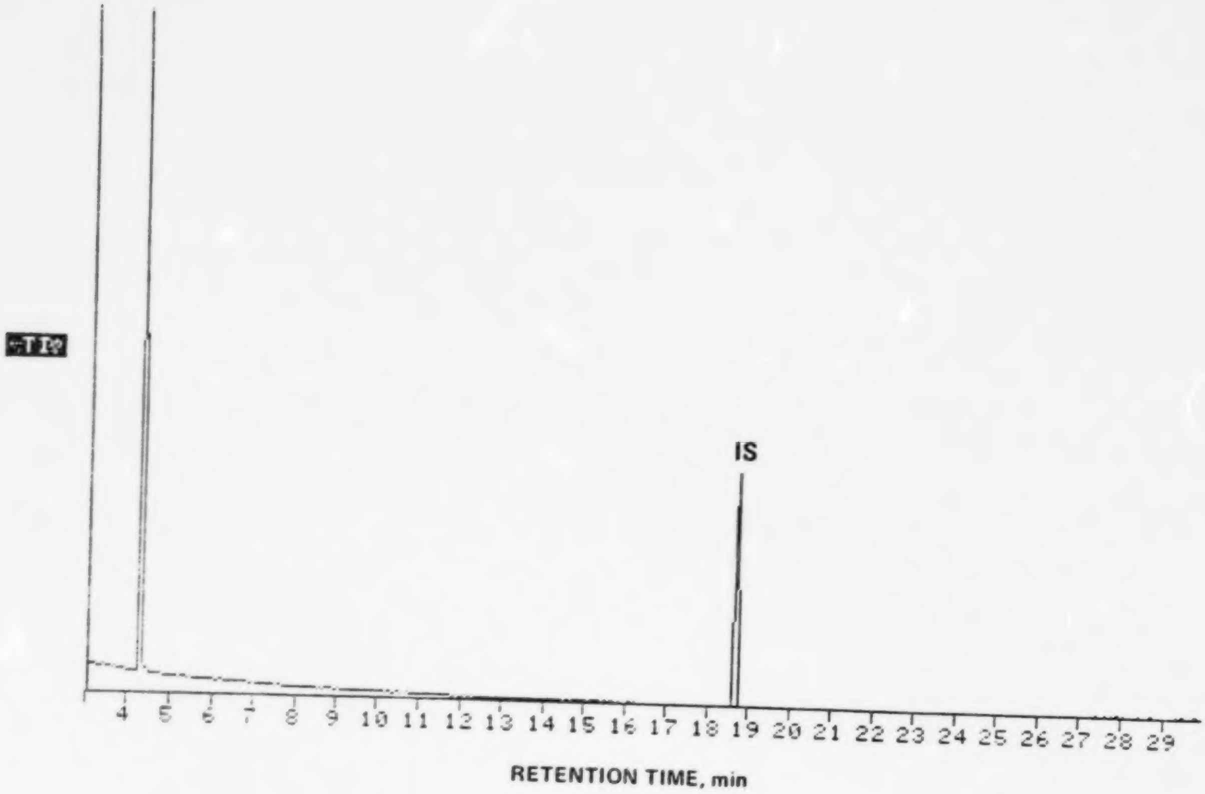
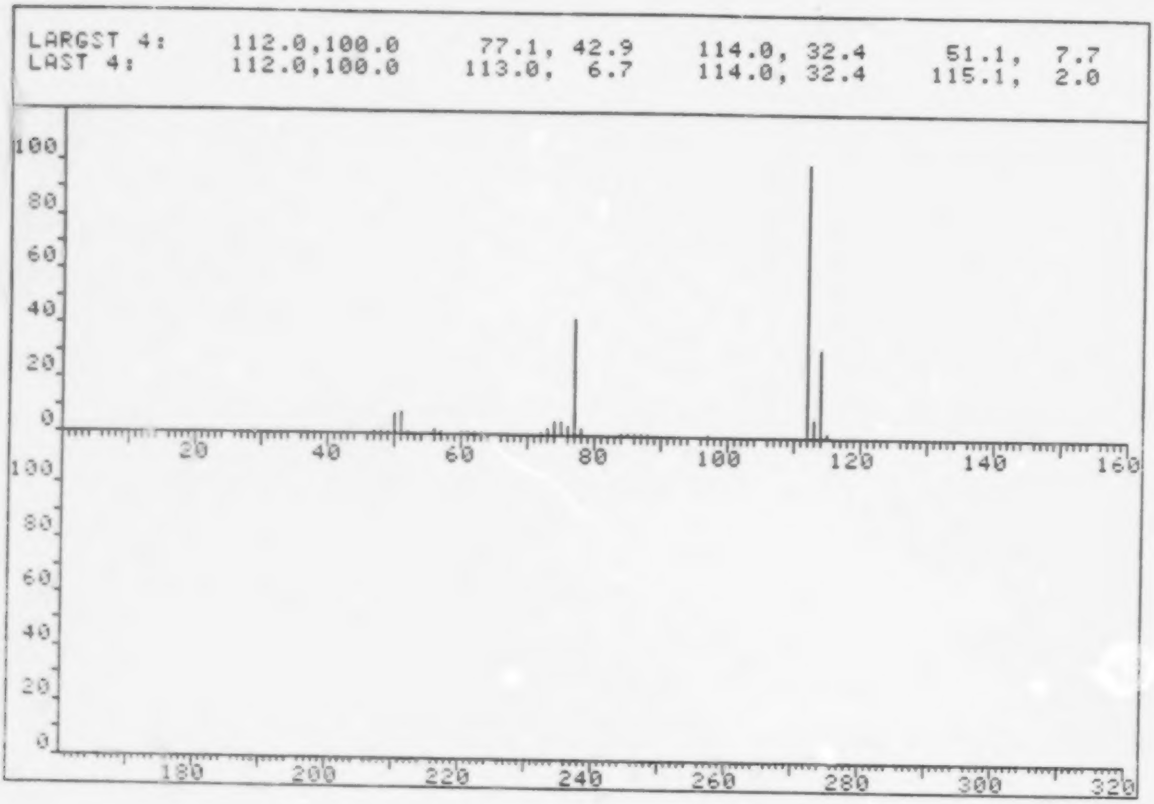


Figure D-22. Chromatogram by GC/MS for chlorobenzene(t_R = 4.3 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal



```
LARGST 4:     112.0,100.0      77.1, 42.9     114.0, 32.4      51.1,  7.7
LAST 4:       112.0,100.0     113.0,  6.7     114.0, 32.4     115.1,  2.0
```

Figure D-23. Mass spectrum for chlorobenzene.

TABLE D-10. KEY IONS AND RELATIVE ABUNDANCES FOR CHLOROBENZENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
50	6.7	75	4.8	112	100.0		
51	5.6	76	4.4	113	7.0		
74	4.4	77	34.4	114	31.5		

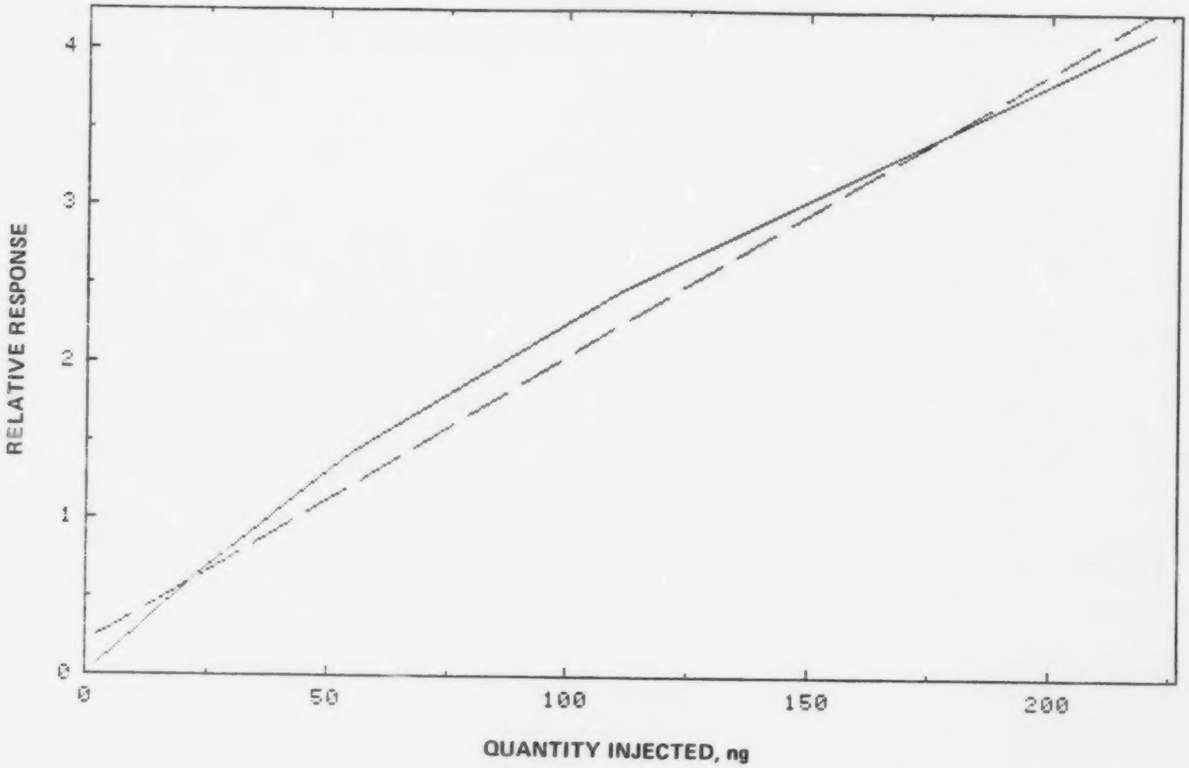


Figure D-24. Calibration curve for the determination of chlorobenzene by GC/MS.

Range: 10 - 220 ng
Correlation coefficient: 0.994
Slope: 1.8×10^{-2}
y-Intercept: 2.1×10^{-1}

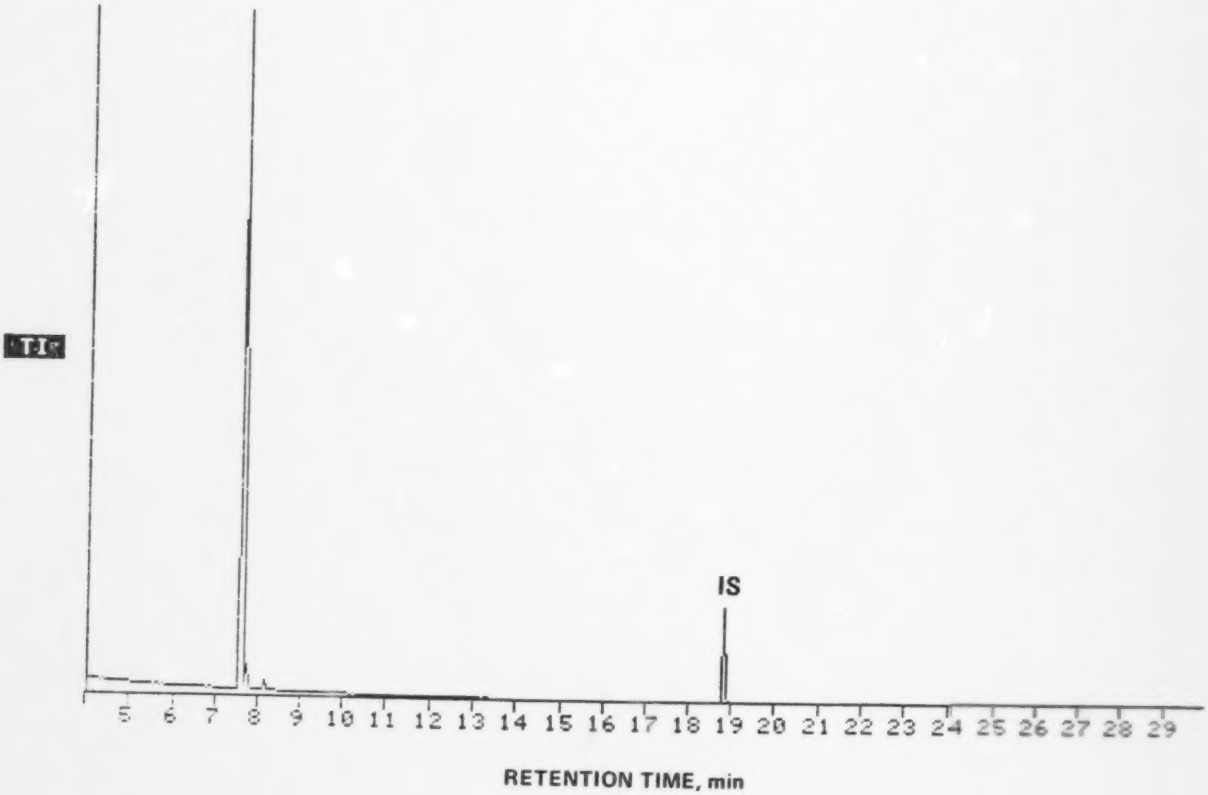
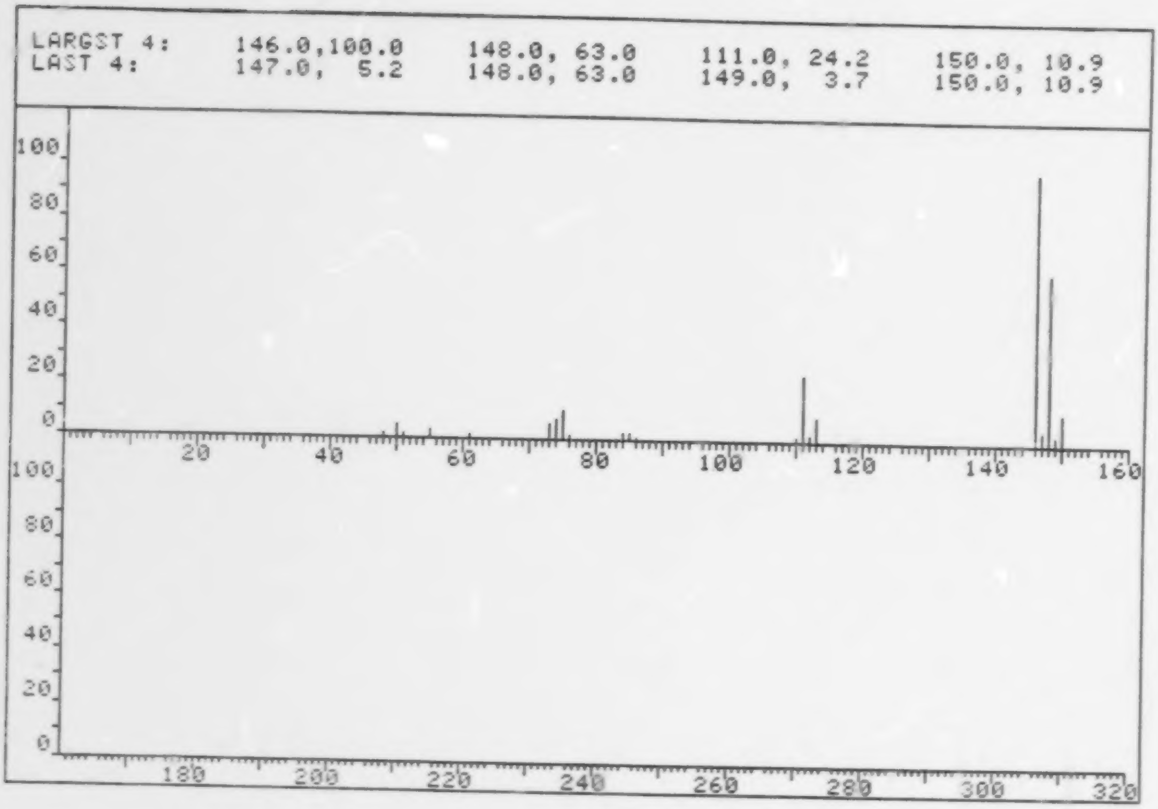


Figure D-25. Chromatogram by GC/MS for m-dichlorobenzene(t_R = 7.7 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal



```
LARGST 4:     146.0,100.0     148.0, 63.0     111.0, 24.2     150.0, 10.9
LAST 4:       147.0,  5.2     148.0, 63.0     149.0,  3.7     150.0, 10.9
```

Figure D-26. Mass spectrum for m-dichlorobenzene.

TABLE D-11. KEY IONS AND RELATIVE ABUNDANCES FOR *m*-DICHLOROBENZENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
48	1.4	74	7.3	110	1.7	148	63.0
50	4.7	75	10.4	111	24.2	149	3.7
51	1.4	76	1.4	112	2.4	150	10.9
55	2.9	84	1.6	113	8.8		
61	1.4	85	2.3	146	100.0		
73	5.6	86	1.0	147	5.2		

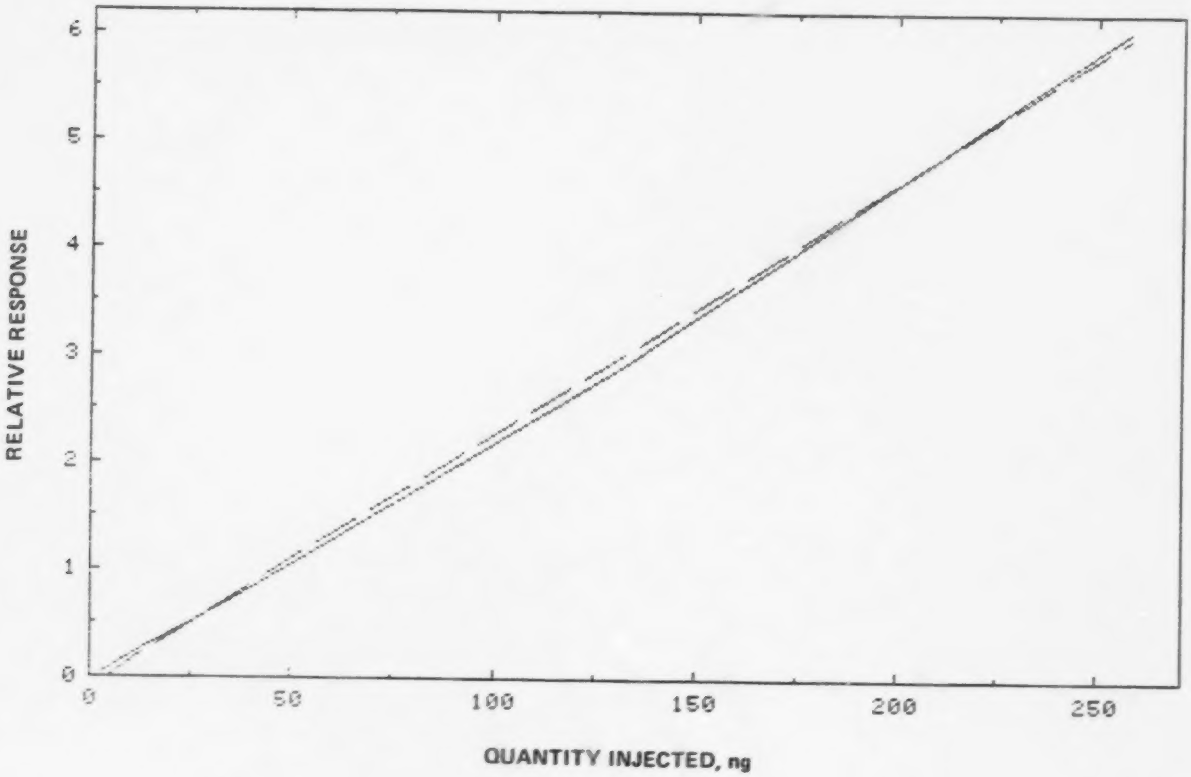


Figure D-27. Calibration curve for the determination of m-dichlorobenzene by GC/MS.

Range: 10 - 260 ng
Correlation coefficient: 0.999
Slope: 1.5×10^{-2}
y-Intercept: 8.7×10^{-2}

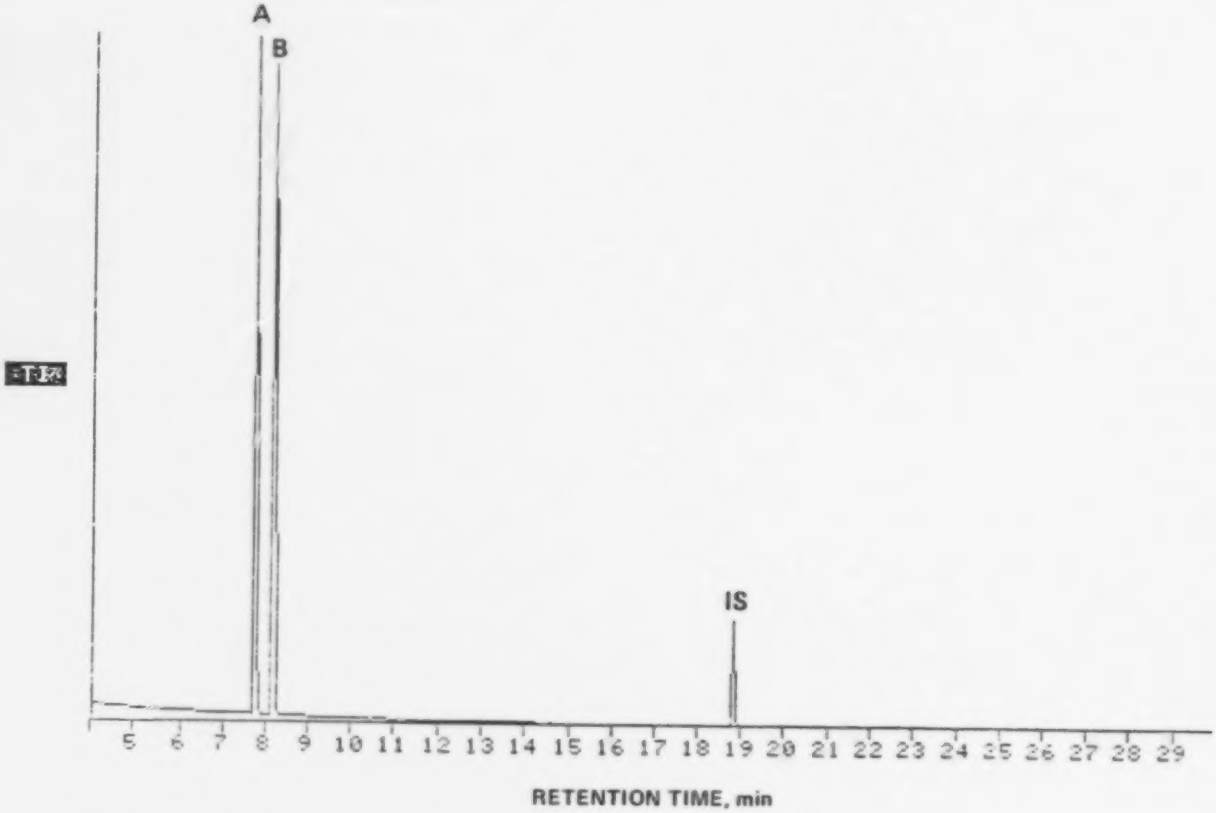
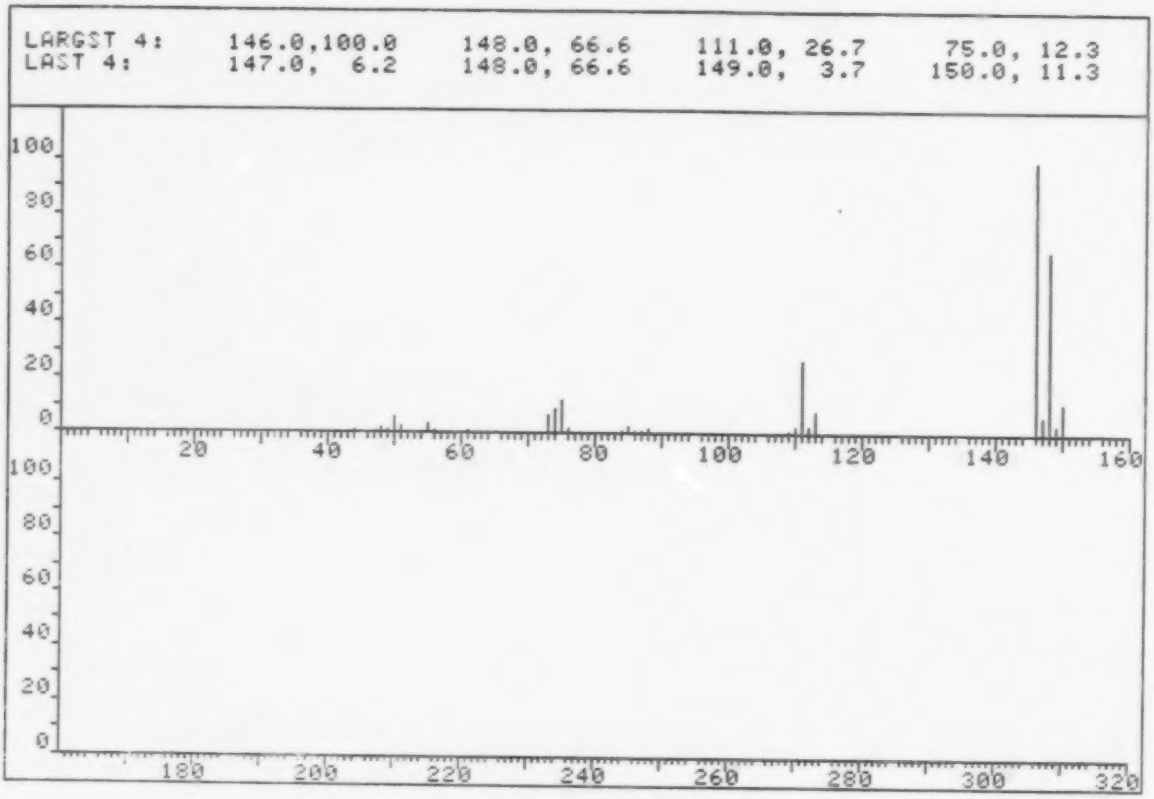


Figure D-28. Chromatogram by GC/MS for

(A) p-dichlorobenzene(t_R = 7.8 min) and
(B) o-dichlorobenzene(t_R = 8.2 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal



```
LARGST 4:    146.0,100.0    148.0, 66.6    111.0, 26.7     75.0, 12.3
LAST 4:      147.0,  6.2    148.0, 66.6    149.0,  3.7    150.0, 11.3
```

Figure D-29. Mass spectrum for p-dichlorobenzene.

TABLE D-12. KEY IONS AND RELATIVE ABUNDANCES FOR p-DICHLOROBENZENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
47	1.3	62	2.4	85	4.9	113	14.3
48	1.8	63	2.2	86	2.0	114	1.2
49	4.6	72	2.1	87	1.9	146	100.0
50	23.9	73	18.8	97	.8	147	7.0
51	6.9	74	28.5	108	.9	148	58.7
55	11.2	75	40.7	109	2.2	149	4.0
56	3.9	76	5.5	110	3.4	150	9.0
60	2.8	83	.8	111	47.0	151	.8
61	3.7	84	3.6	112	4.5		

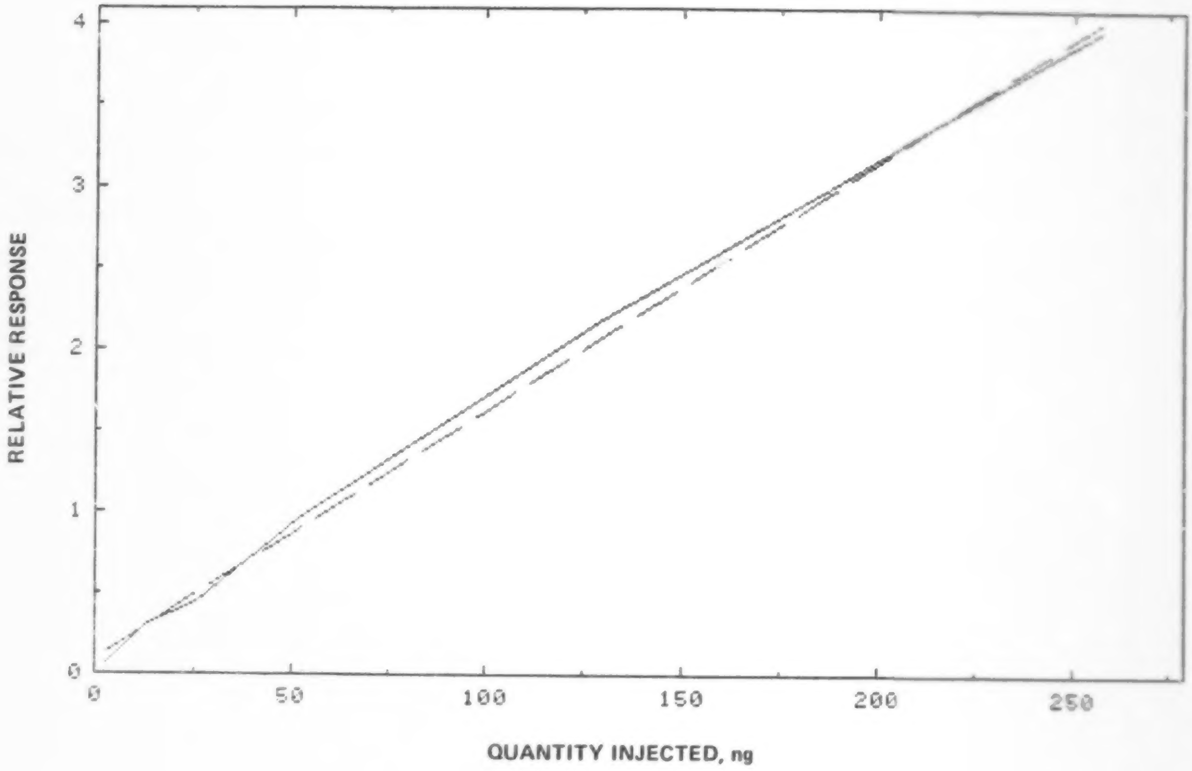
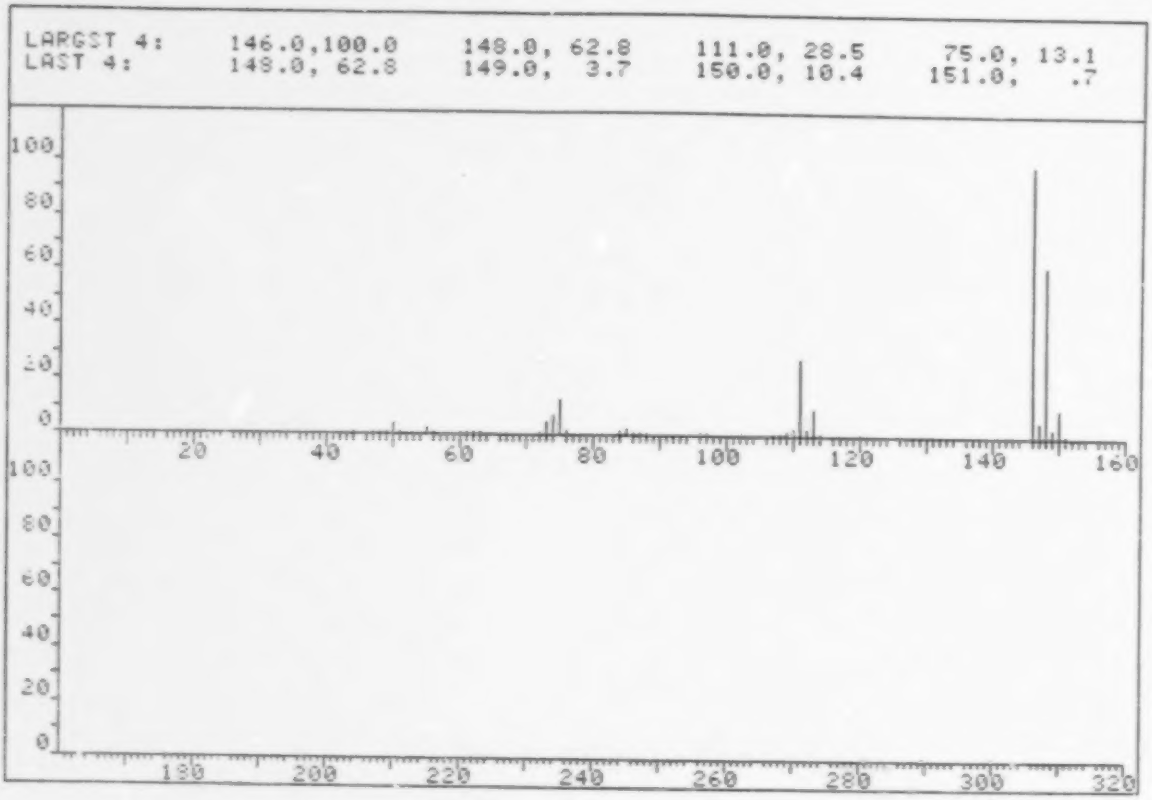


Figure D-30. Calibration curve for the determination of p-dichlorobenzene by GC/MS.

Range: 10 - 260 ng
Correlation coefficient: 0.999
Slope: 1.3×10^{-2}
y-Intercept: 6.7×10^{-2}



```
LARGST 4:      146.0,100.0     148.0, 62.8     111.0, 28.5      75.0, 13.1
LAST 4:        148.0, 62.8     149.0,  3.7     150.0, 10.4     151.0,    .7
```

Figure D-31. Mass spectrum for o-dichlorobenzene.

TABLE D-13. KEY IONS AND RELATIVE ABUNDANCES FOR o-DICHLOROBENZENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
47	1.0	62	1.4	86	2.0	112	3.8
48	.7	63	1.3	87	1.5	113	12.9
49	3.2	72	2.0	96	.5	114	.8
50	13.4	73	10.9	97	.8	146	100.0
51	4.1	74	17.3	107	.6	147	6.7
55	5.6	75	29.0	108	.6	148	59.1
56	2.1	76	3.9	109	2.1	149	4.1
60	1.4	84	4.3	110	3.3	150	9.6
61	2.4	85	4.5	111	41.5	151	.7

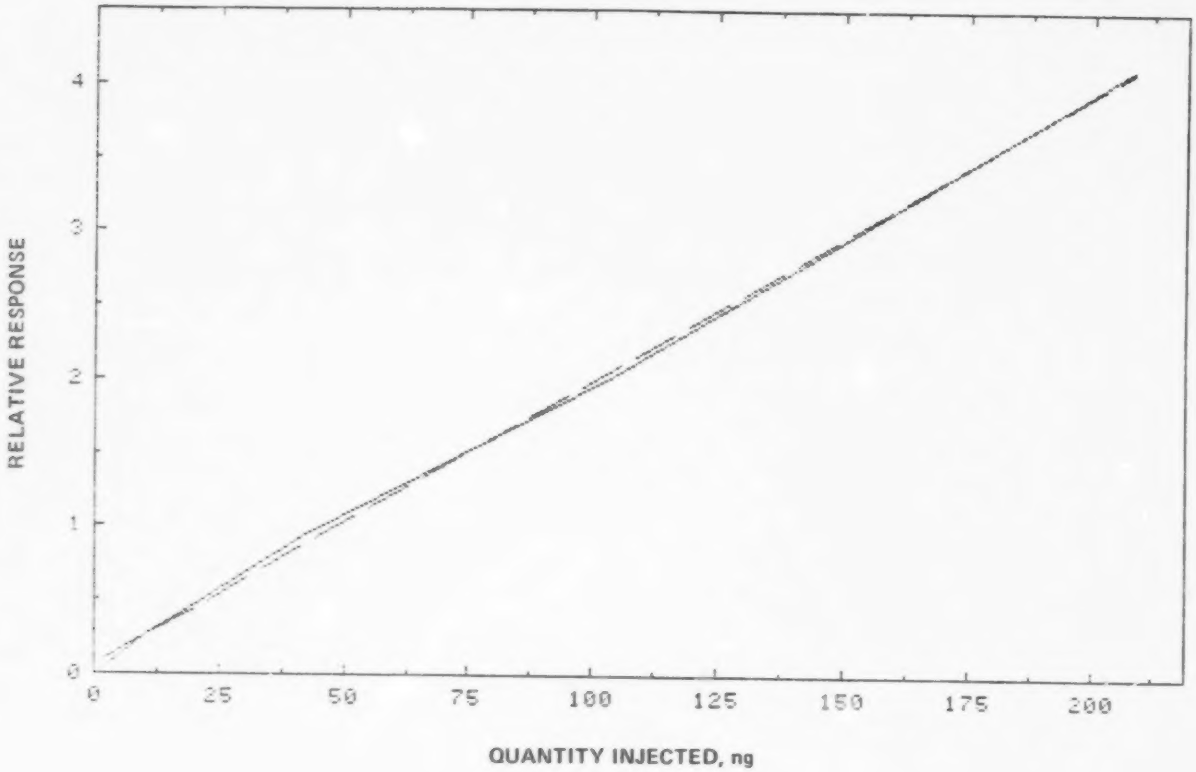


Figure D-32. Calibration curve for the determination of o-dichlorobenzene by GC/MS.

Range: 10 - 210 ng
Correlation coefficient: 0.999
Slope: 1.9×10^{-2}
y-Intercept: 6.6×10^{-2}

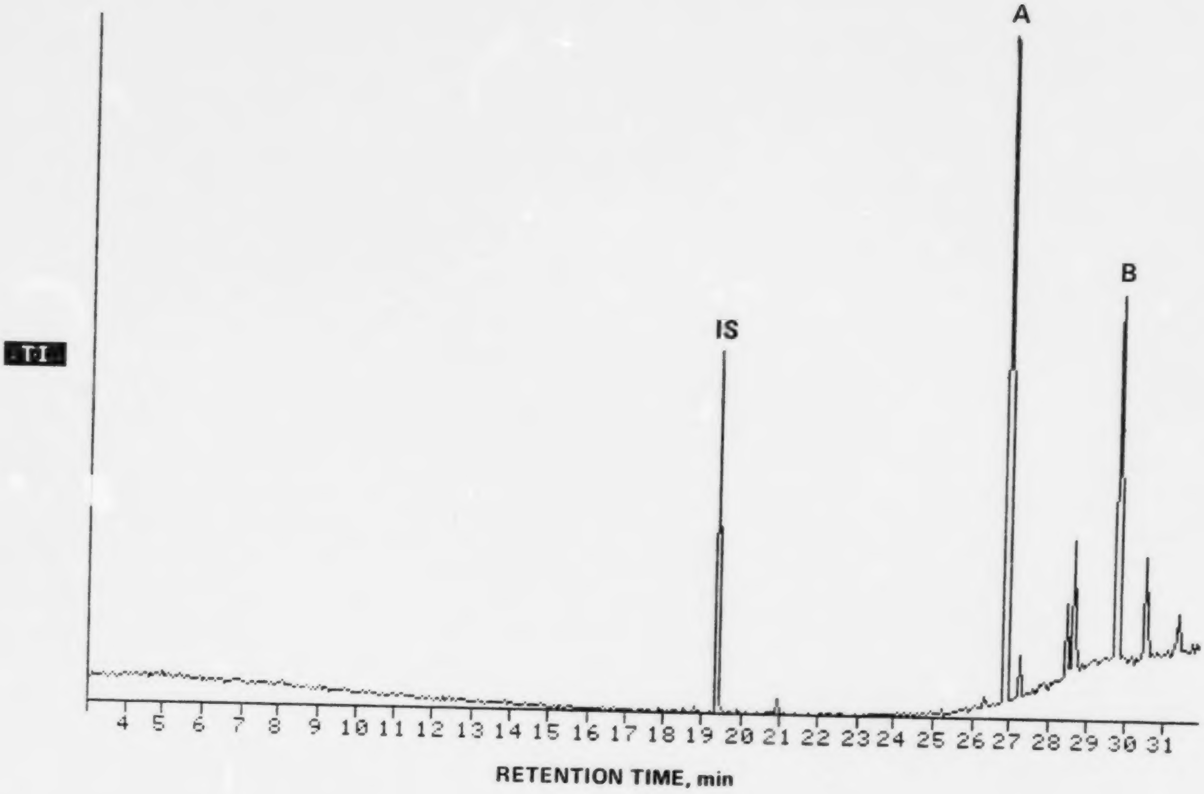
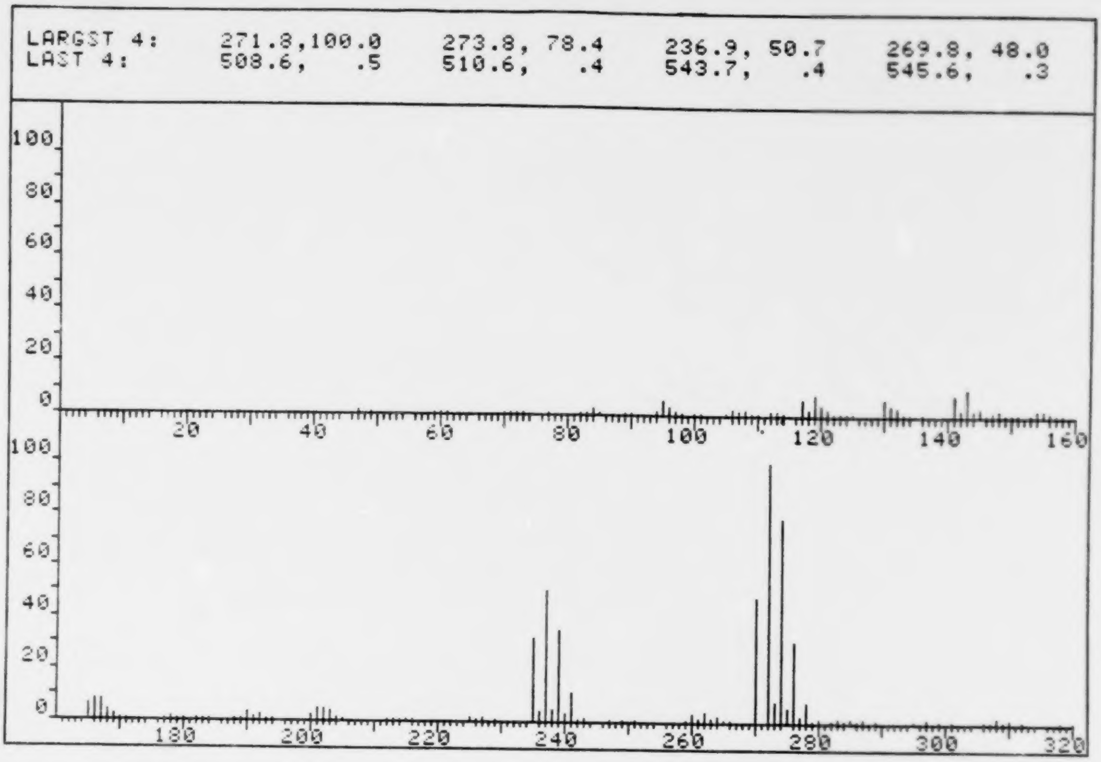


Figure D-33. Chromatogram by GC/MS for

(A) 1,3,4-metheno-1*H*-cyclobuta[cd]pentalene, 1,1a,2,2,3,3a,4,5,5,5a,5b,6- dodecachloro-octahydro- (t_R = 26.9 min) and
(B) hexabromobiphenyl(t_R = 29.7 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal



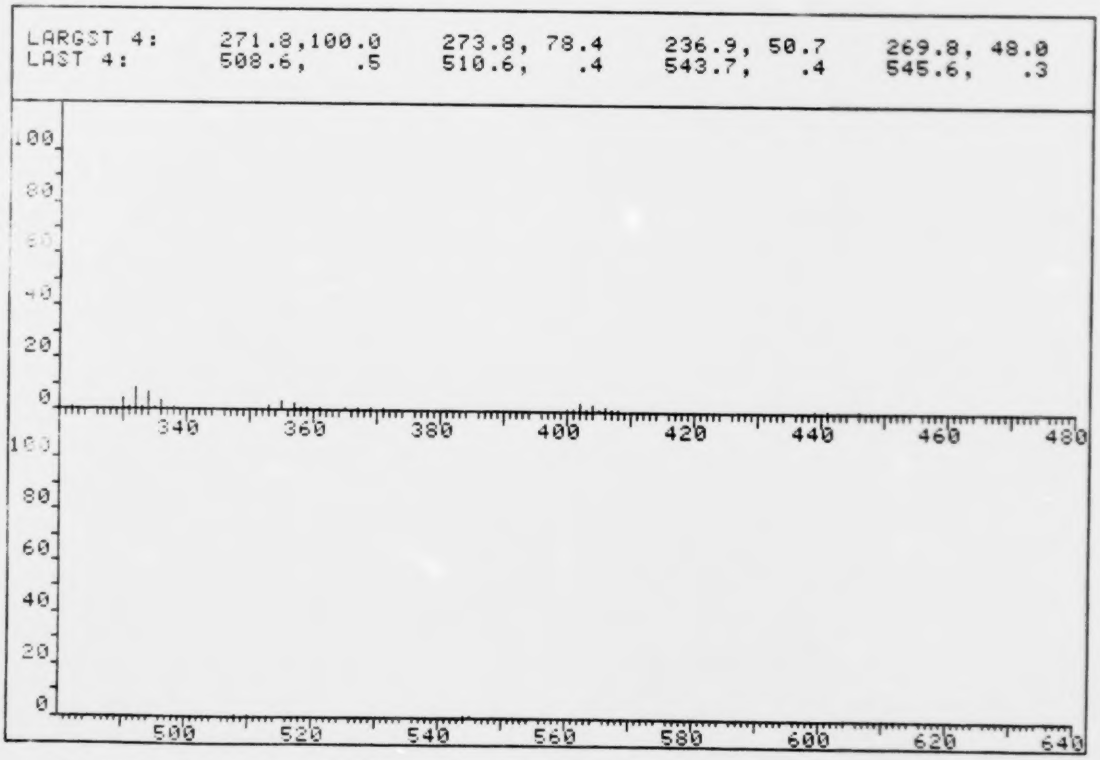


Figure D-34. Mass spectrum for 1,3,4-metheno-1H-cyclobuta[cd]pentalene, 1,1a,2,2,3,3a,4,5,5,5a,5b,6- dodecachlorooctahydro- .

TABLE D-14. KEY IONS AND RELATIVE ABUNDANCES FOR 1,3,4-METHENO-1H-CYCLOBUTA[cd]-PENTALENE, 1,1a,2,2,$\overline{3}$,3a,4,5,5,5a,6-DODECACHLOROOCTAHYDRO-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
47	1.3	146	.6	214	.6	286	.7
60	.9	147	1.5	215	.5	287	1.1
71	.8	148	2.1	225	1.5	295	.7
72	.5	149	.9	227	1.0	297	1.0
77	.5	150	.7	229	.5	299	.7
82	.8	153	1.1	235	32.1	306	.8
83	.7	154	2.4	236	4.0	308	1.8
84	2.2	155	2.2	237	50.7	310	1.3
94	1.5	156	1.7	238	5.0	312	.7
95	5.7	157	.7	239	35.2	320	.8
96	3.3	165	6.0	240	2.9	322	.6
97	1.3	166	7.5	241	10.7	330	4.2
106	2.3	167	7.8	242	.9	331	.7
107	1.5	168	3.5	243	1.7	332	8.2
108	2.7	169	1.8	259	.6	333	1.3
112	1.8	177	.8	260	2.4	334	6.6
113	1.8	178	1.2	261	1.0	335	.9
114	.5	179	.6	262	3.7	336	3.0
117	6.4	180	.8	263	1.1	338	.7
118	2.5	183	.5	264	1.8	353	1.4
119	8.5	184	.5	270	48.0	355	3.2
120	4.0	188	.6	272	100.0	357	2.5
121	2.8	189	.9	273	7.9	359	1.3
122	.5	190	3.3	274	78.4	365	.5
130	6.3	191	1.1	275	5.4	367	1.0
131	4.5	192	2.3	276	31.2	369	1.1
132	3.6	193	.7	277	2.2	371	.5
133	.9	194	.5	278	7.6	400	1.0
134	.8	200	1.8	279	.5	402	2.8
141	8.4	201	4.6	280	1.1	404	2.8
142	2.7	202	4.7	282	.5	405	.6
143	10.7	203	3.7	283	1.1	406	1.8
144	2.2	204	1.3	284	.7	408	.8
145	3.5	205	.6	285	1.5	508	.5

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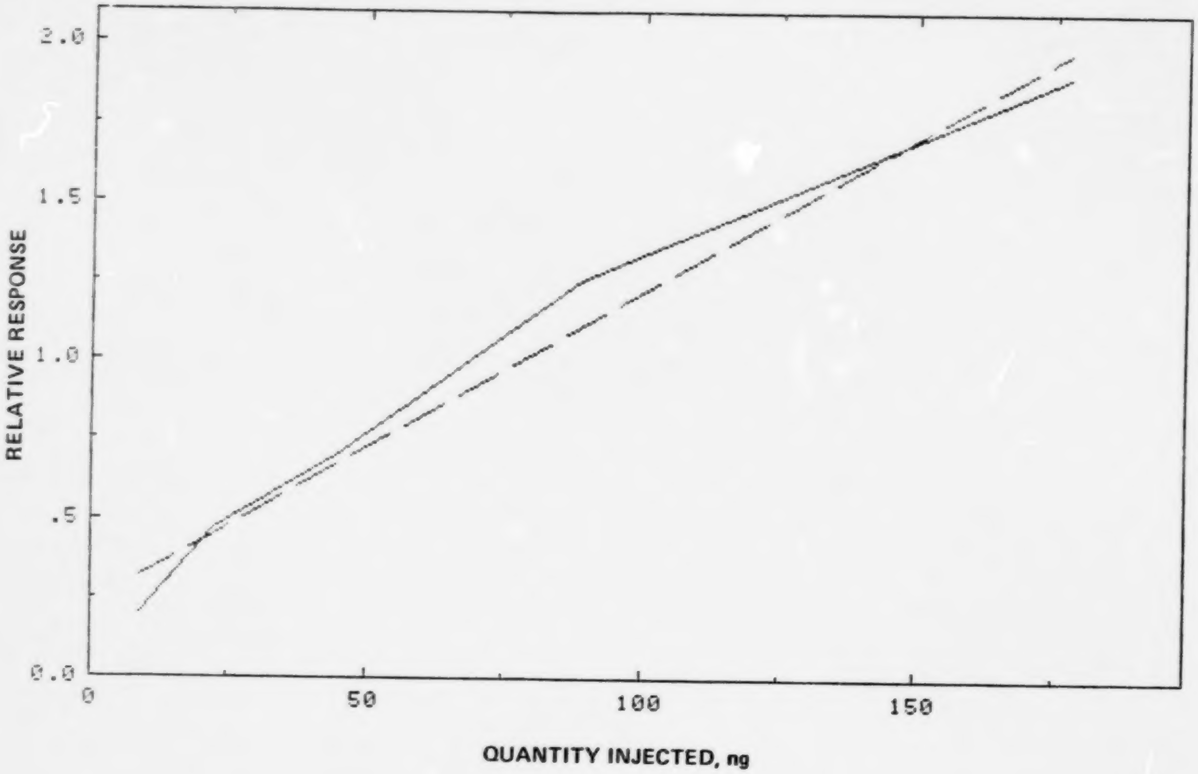


Figure D-35. Calibration curve for the determination of 1,3,4-metheno-1$\underline{H}$-cyclobuta[cd]pentalene, 1,1a,2,2,3,3a,4,5,5,5a,5b,6-dodecachloro-octahydro- by GC/MS.

Range: 9 - 180 ng
Correlation coefficient: 0.989
Slope: 9.8×10^{-3}
y-Intercept: 2.3×10^{-1}

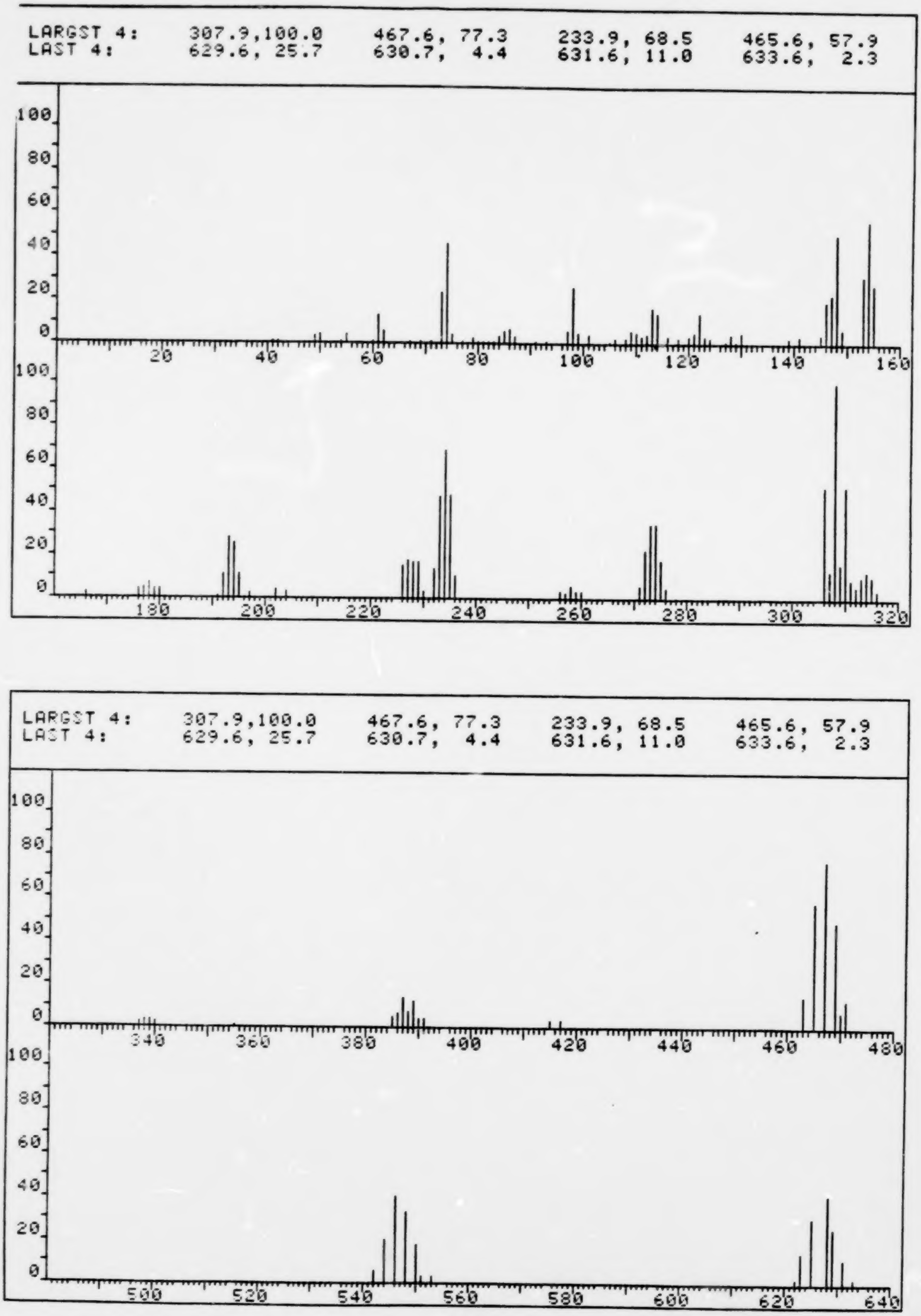


Figure D-36. Mass spectrum for hexabromobiphenyl.

TABLE D-15. KEY IONS AND RELATIVE ABUNDANCES FOR HEXABROMOBIPHENYL

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	1.2	114	13.8	226	15.2	340	2.6
49	2.8	116	3.5	227	17.1	385	4.7
50	4.2	118	2.6	228	16.4	386	6.5
54	.9	120	3.0	229	16.1	387	14.0
55	4.2	121	5.1	230	2.8	388	7.2
60	.7	122	14.3	232	13.5	389	12.4
61	12.3	123	3.7	233	47.0	390	4.0
62	5.4	124	2.3	234	68.5	391	4.2
67	.9	128	4.2	235	47.7	415	2.8
69	.9	129	.7	236	10.5	417	2.8
71	.7	130	4.7	256	2.8	463	14.5
73	23.4	139	2.6	257	2.3	465	57.9
74	45.6	141	3.3	258	5.6	467	77.3
75	4.4	145	4.4	259	2.6	469	14.7
79	2.3	146	19.4	260	2.6	469	49.1
80	.7	147	22.9	271	5.1	470	7.0
81	1.2	148	50.2	272	22.2	471	13.1
82	.9	149	6.8	273	34.1	542	5.1
83	.7	153	31.5	274	34.1	544	19.9
84	3.3	154	57.2	275	17.1	546	39.7
85	5.6	155	27.3	276	4.7	548	32.7
86	6.3	166	2.3	306	52.3	550	4.7
87	3.3	176	4.2	307	12.4	550	17.1
97	5.8	177	4.4	308	100.0	551	3.0
98	25.5	178	7.0	309	15.4	553	2.8
99	4.9	179	4.2	310	51.9	622	2.3
101	4.0	180	3.7	311	8.9	623	14.3
105	.7	191	.7	312	5.1	625	30.4
106	2.3	192	11.0	313	10.5	628	40.9
108	2.6	193	28.3	314	13.1	629	25.7
109	5.6	194	25.0	315	10.0	631	4.4
110	5.1	195	10.7	316	3.7	631	11.0
111	3.7	197	2.3	337	2.6	633	2.3
112	4.0	202	4.0	338	2.8		
113	16.4	204	3.0	339	2.8		

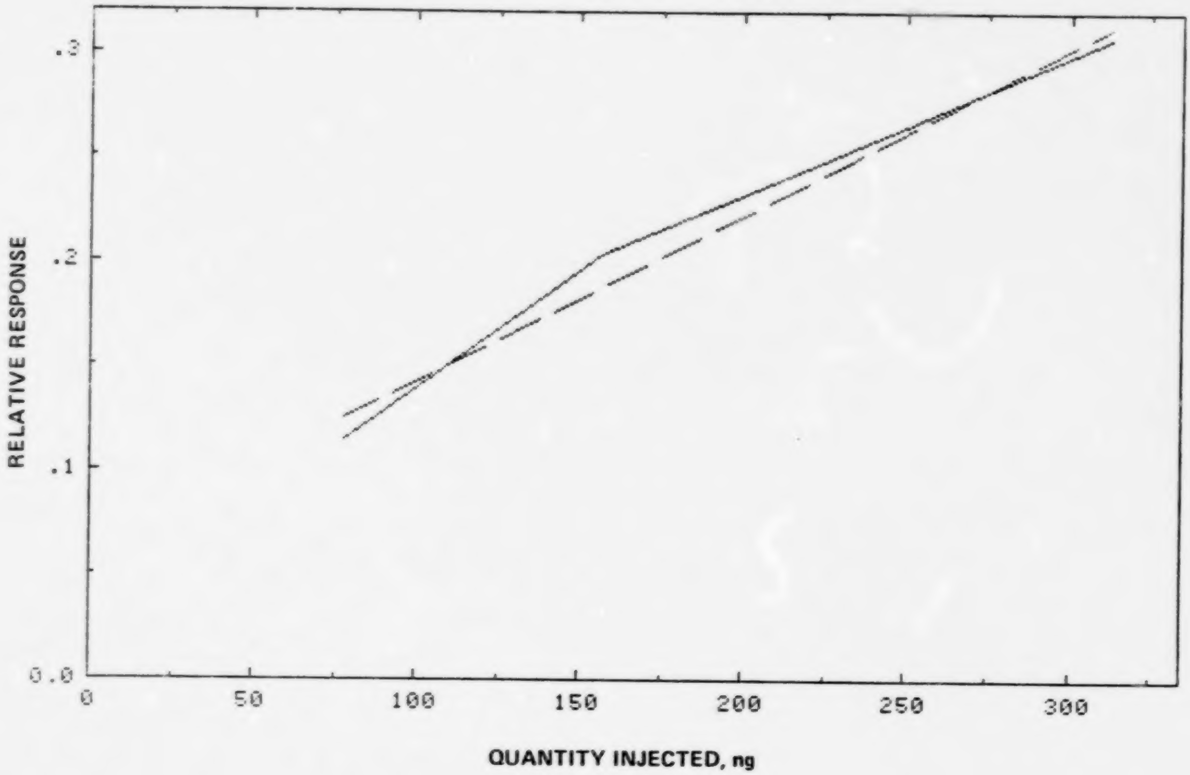


Figure D-37. Calibration curve for the determination of hexabromobiphenyl by GC/MS.

Range: 78 - 310 ng
Correlation coefficient: 0.990
Slope: 8.0×10^{-4}
y-Intercept: 6.3×10^{-2}

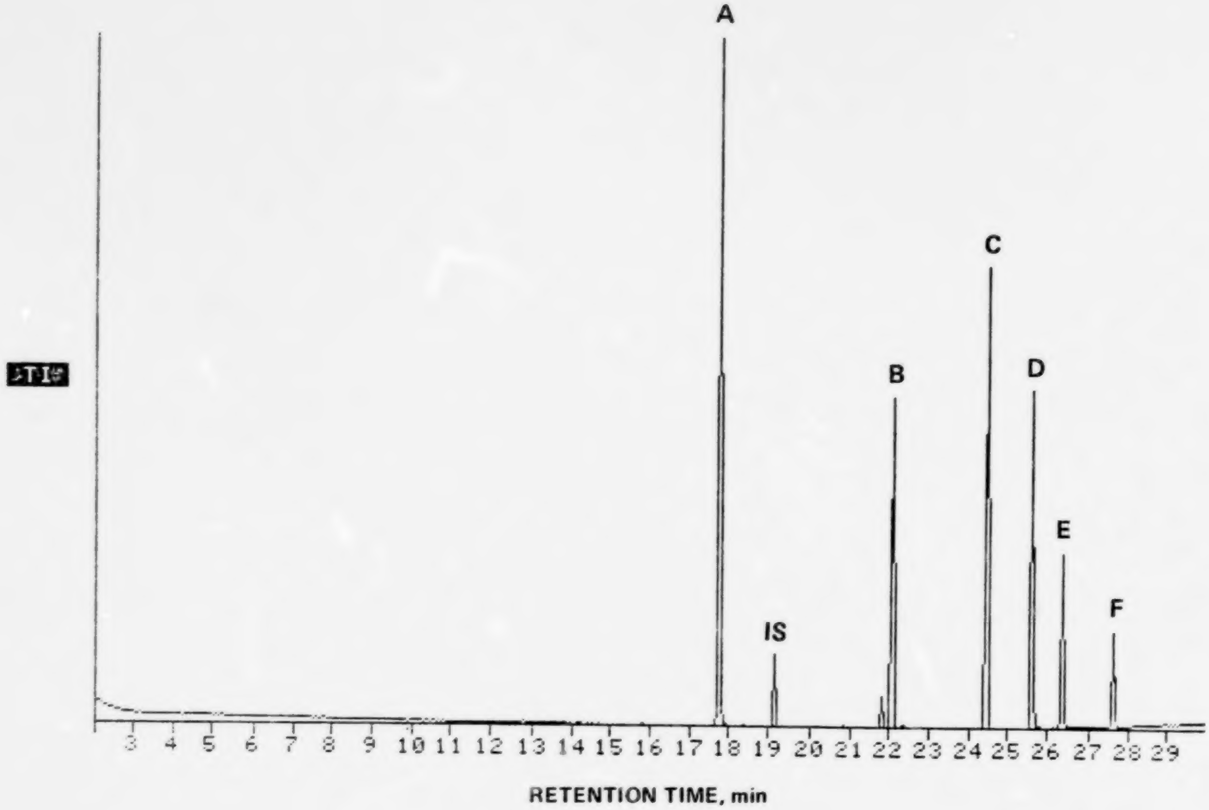


Figure D-38. Chromatogram by GC/MS for

(A) phosphoric acid, dimethyl ester, ester with (*E*)-3-hydroxy-*N*,*N*-dimethylcrotonamide(t_R = 17.8 min),
(B) phosphoric acid, 2-chloro-1-(2,4-dichlorophenyl) vinyl diethyl ester(tR = 22.1 min),
(C) phosphorodithioic acid, *S*-(((*p*-chlorophenyl)thio)methyl) *O*,*O*-diethyl ester(t_R = 24.5 min),
(D) phosphorodithioic acid, *O*,*O*-dimethyl ester, *S*-ester with *N*-(mercaptomethyl)phthalimide(t_R = 25.7 min),
(E) phosphonothioic acid, phenyl-, *O*-(4-bromo-2,5-dichlorophenyl) *O*-methyl ester(t_R = 26.4 min), and
(F) coumarin, 3-chloro-7-hydroxy-4-methyl-, *O*-ester with *O*,*O*-diethyl phosphorothioate(t_R = 27.7 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

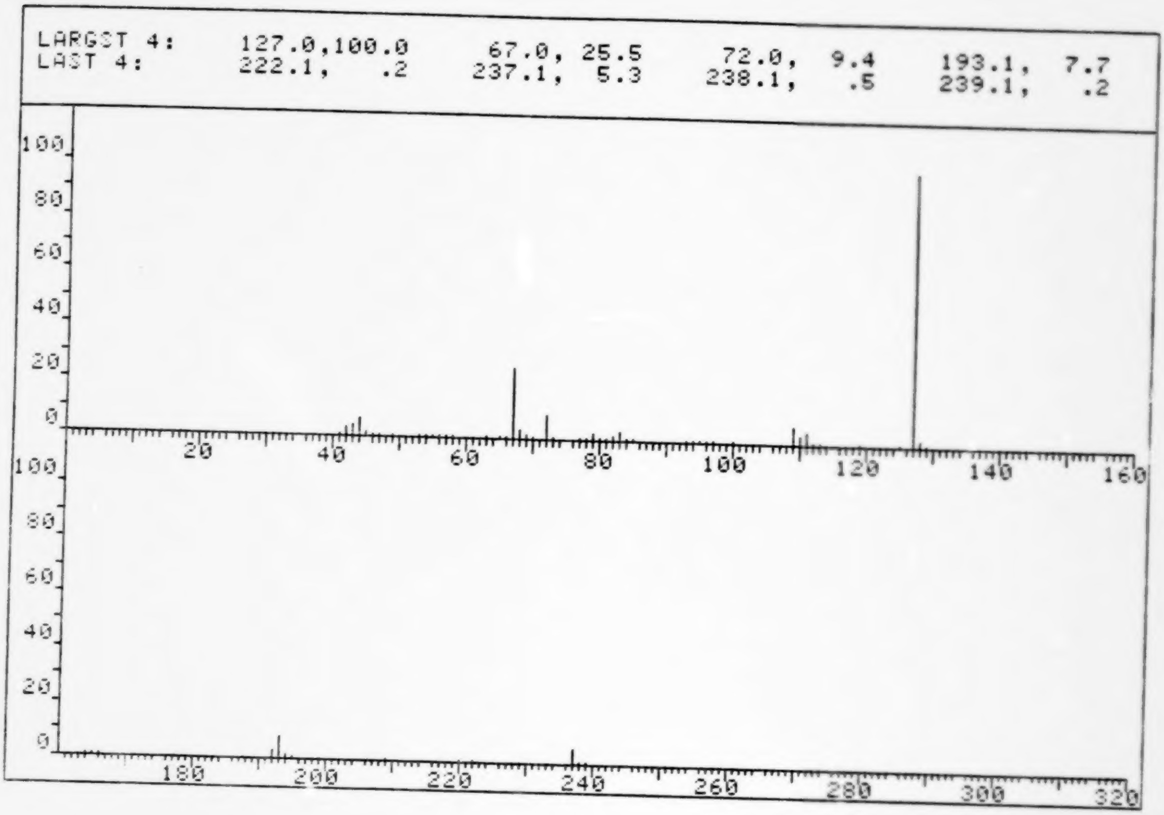


Figure D-39. Mass spectrum for phosphoric acid, dimethyl ester, ester with (E)-3-hydroxy-N,N-dimethylcrotonamide.

TABLE D-16. KEY IONS AND RELATIVE ABUNDANCES FOR PHOSPHORIC ACID, DIMETHYL ESTER, ESTER WITH (*E*)-3-HYDROXY-*N*,*N*-DIMETHYLCROTONAMIDE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	.6	66	1.3	95	1.2	129	.9
42	3.7	67	25.5	96	1.0	164	.9
43	4.0	68	3.3	109	6.1	192	3.5
44	6.6	69	1.6	110	3.6	193	7.7
45	1.6	72	9.4	111	5.4	194	1.6
46	.6	79	2.7	112	.8	237	5.3
47	.9	82	1.5	113	.6	238	.5
55	1.1	83	3.2	125	.5		
56	.9	84	.5	127	100.0		
58	.6	93	.7	128	2.4		

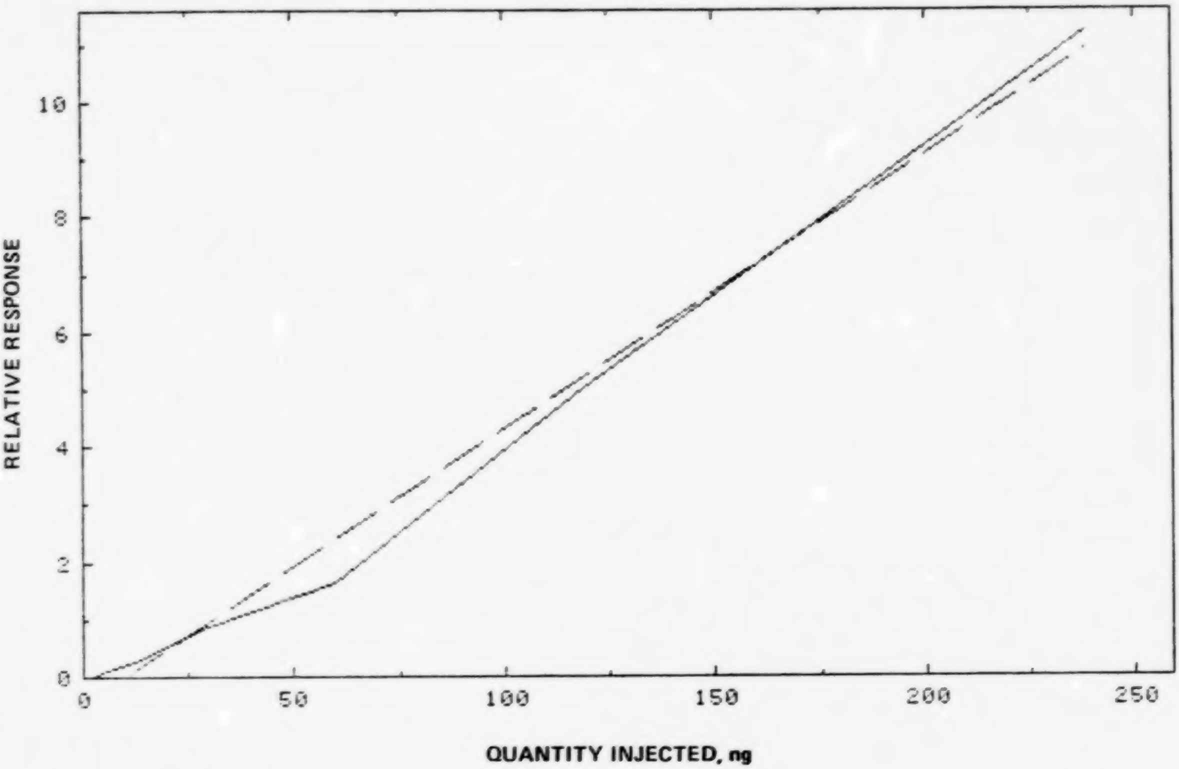
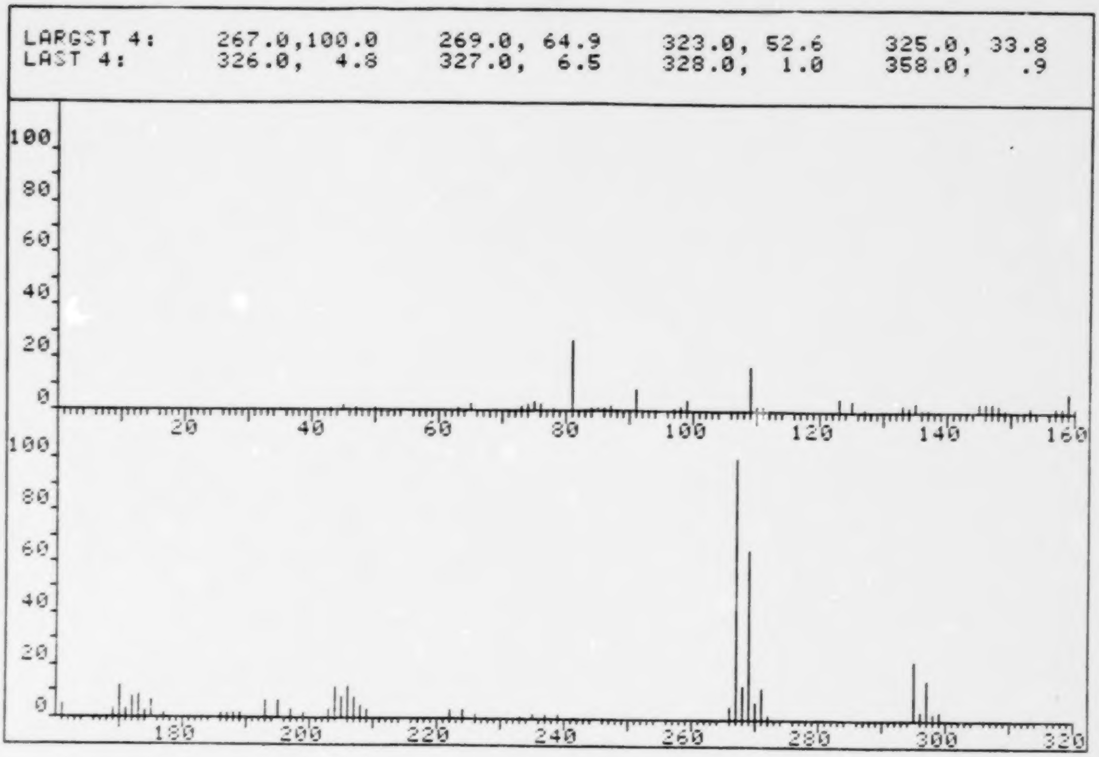
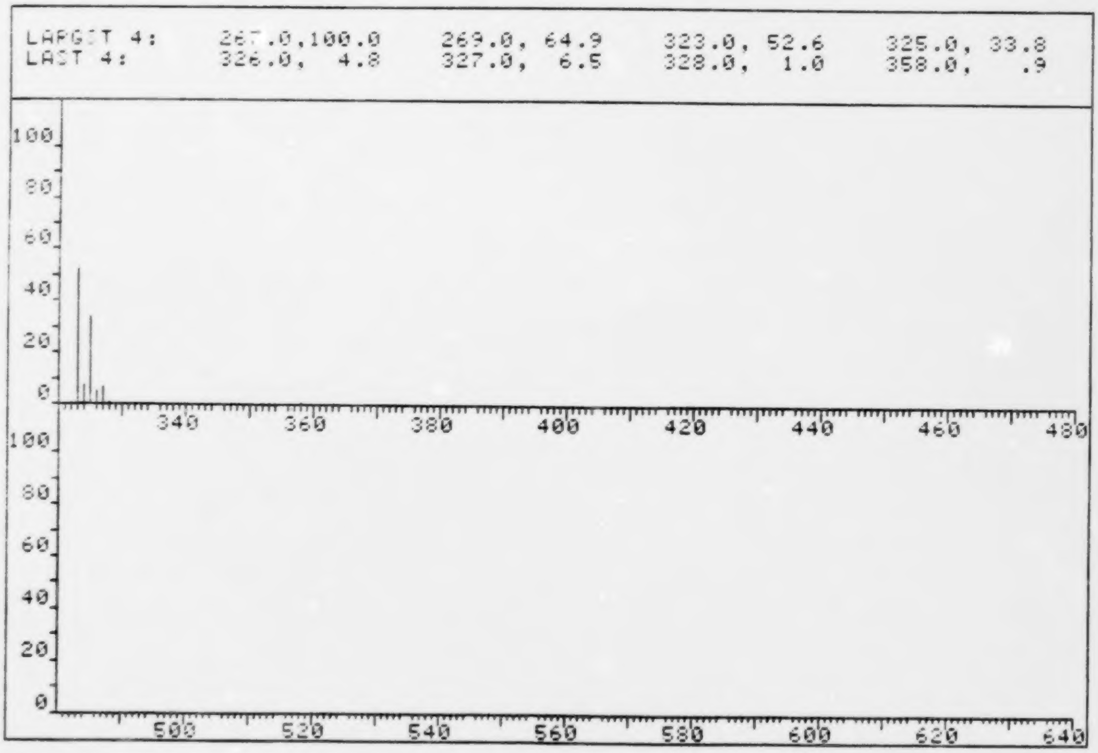


Figure D-40. Calibration curve for the determination of phosphoric acid, dimethyl ester, ester with (E)-3-hydroxy-N,N-dimethylcrotonamide by GC/MS.

Range: 30 - 240 ng
Correlation coefficient: 0.996
Slope: 4.8×10^{-2}
y-Intercept: -4.6×10^{-1}



```
LARGST 4:     267.0,100.0     269.0, 64.9     323.0, 52.6     325.0, 33.8
LAST 4:       326.0,   4.8    327.0,   6.5    328.0,   1.0    358.0,    .9
```



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LARGST 4:     267.0,100.0     269.0, 64.9     323.0, 52.6     325.0, 33.8
LAST 4:       326.0,   4.8    327.0,   6.5    328.0,   1.0    358.0,    .9
```

Figure D-41. Mass spectrum for phosphoric acid, 2-chloro-1-(2,4-dichlorophenyl) vinyl diethyl ester.

TABLE D-17. KEY IONS AND RELATIVE ABUNDANCES FOR PHOSPHORIC ACID, 2-CHLORO-1-(2,4-DICHLOROPHENYL) VINYL DIETHYL ESTER

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
43	1.0	123	4.6	173	8.8	235	1.4
45	1.7	125	4.2	174	2.1	237	1.1
47	.9	127	1.1	175	6.1	239	1.0
50	1.0	133	2.4	177	1.4	266	4.7
63	1.1	134	1.5	186	1.4	267	100.0
65	2.1	135	3.0	187	1.4	268	12.2
73	1.4	136	1.0	188	1.0	269	64.9
74	2.7	137	1.0	189	1.0	270	6.5
75	3.1	145	3.8	193	5.9	271	11.5
76	2.5	146	3.5	195	6.0	272	1.0
78	.9	147	2.8	197	2.5	295	21.8
81	26.6	148	2.1	199	1.0	296	3.1
84	1.0	149	.8	203	2.6	297	15.1
85	1.0	153	1.4	204	11.4	298	2.2
86	.9	157	1.6	205	7.7	299	2.4
87	1.3	158	1.6	206	11.5	323	52.6
91	8.4	159	6.9	207	8.0	324	6.9
97	1.0	160	1.5	208	4.3	325	33.8
98	1.8	161	4.3	209	2.6	326	4.8
99	4.5	169	2.9	222	2.5	327	6.5
109	17.0	170	11.5	224	2.5	328	1.0
110	1.3	171	2.5	226	1.0	358	.9
111	1.9	172	8.0	233	.9		

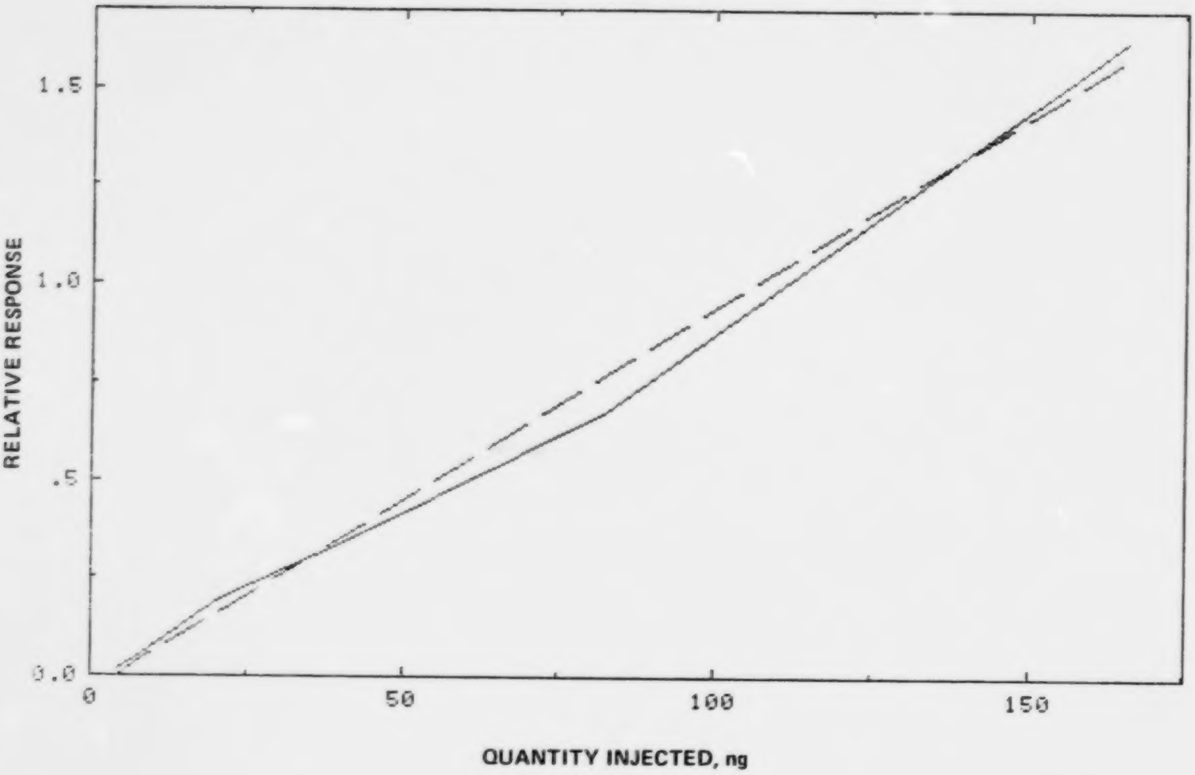


Figure D-42. Calibration curve for the determination of phosphoric acid, 2-chloro-1-(2,4-dichlorophenyl) vinyl diethyl ester by GC/MS.

Range: 20 - 170 ng
Correlation coefficient: 0.997
Slope: 9.8×10^{-3}
y-Intercept: -3.8×10^{-2}

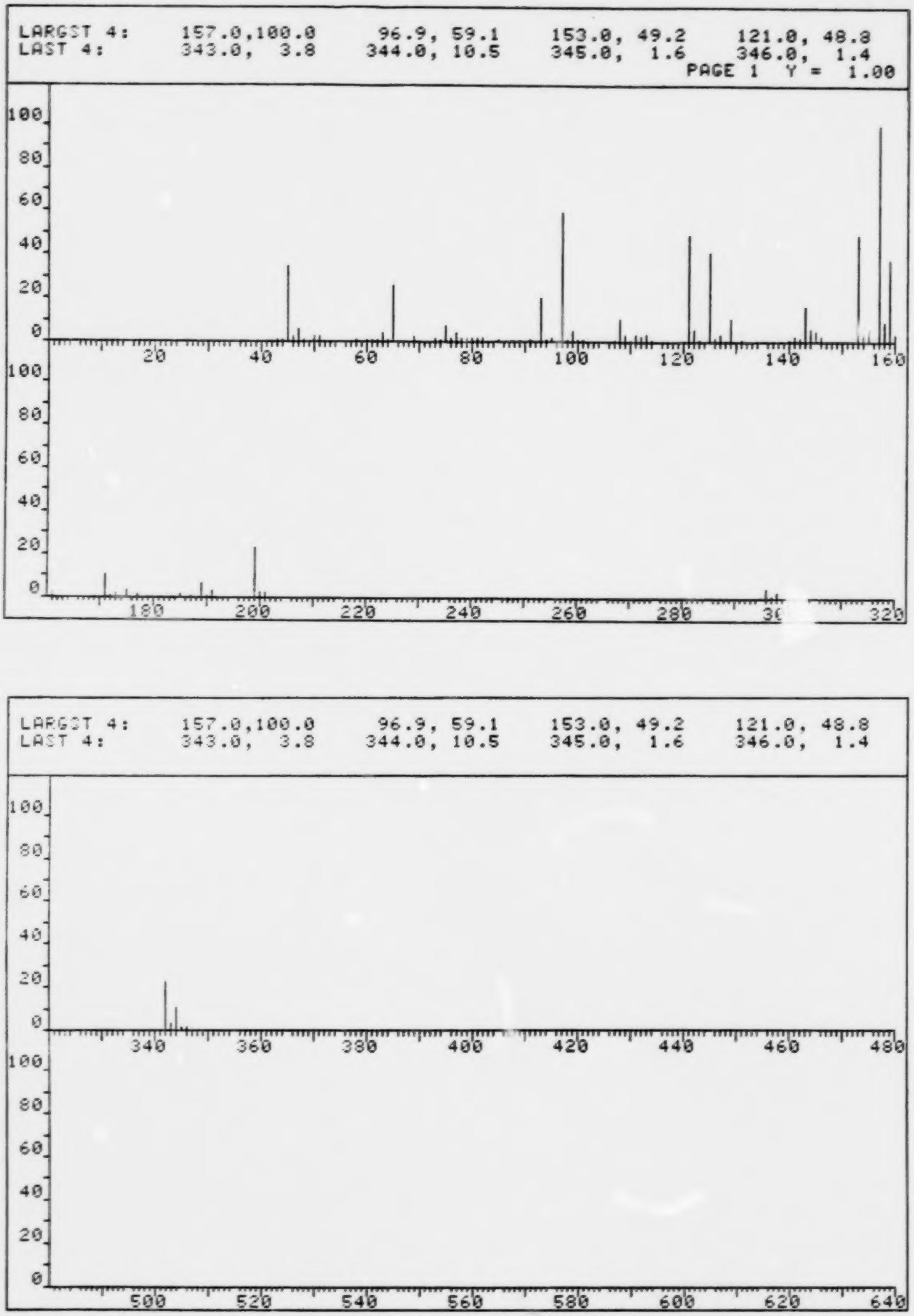


Figure D-43. Mass spectrum for phosphorodithioic acid, S-(((p-chlorophenyl)thio)methyl) O,O-diethyl ester.

TABLE D-18. KEY IONS AND RELATIVE ABUNDANCES FOR PHOSPHORODITHIOIC ACID, S-(((*p*-CHLOROPHENYL)THIO)METHYL), *O*,*O*-DIETHYL ESTER

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
43	1.2	80	1.6	122	5.4	171	9.9
45	34.9	81	1.8	123	.9	172	.7
46	2.7	82	1.8	125	40.8	173	1.2
47	5.9	85	.9	126	1.5	175	2.8
48	.8	91	.8	127	3.5	177	1.1
50	2.2	93	19.9	129	10.6	185	1.1
51	2.0	94	.8	131	1.1	187	.8
58	.9	95	1.5	140	.8	189	5.8
60	.7	97	59.1	141	2.2	190	.7
61	1.1	98	.8	142	1.3	191	2.5
62	.7	99	5.1	143	15.9	199	23.1
63	4.4	100	.8	144	5.7	200	1.9
64	1.1	101	.8	145	5.3	201	2.0
65	25.6	107	1.1	146	2.3	296	4.1
69	2.6	108	10.7	153	49.2	297	.8
73	1.5	109	3.6	154	7.5	298	2.0
74	1.2	110	1.1	155	5.9	342	22.8
75	7.1	111	3.0	157	100.0	343	3.8
76	1.5	112	2.3	158	9.2	344	10.5
77	4.5	113	3.5	159	37.7	345	1.6
78	1.7	114	.7	160	3.2	346	1.4
79	1.8	121	48.8	161	1.7		

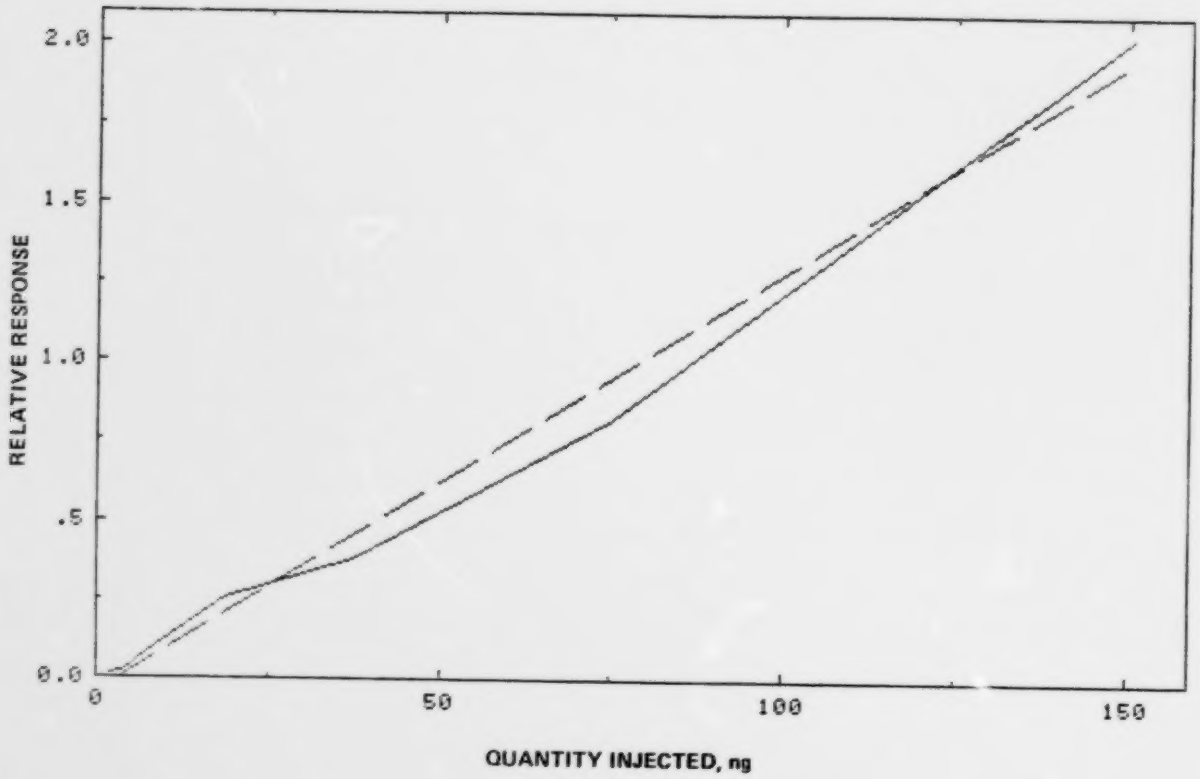
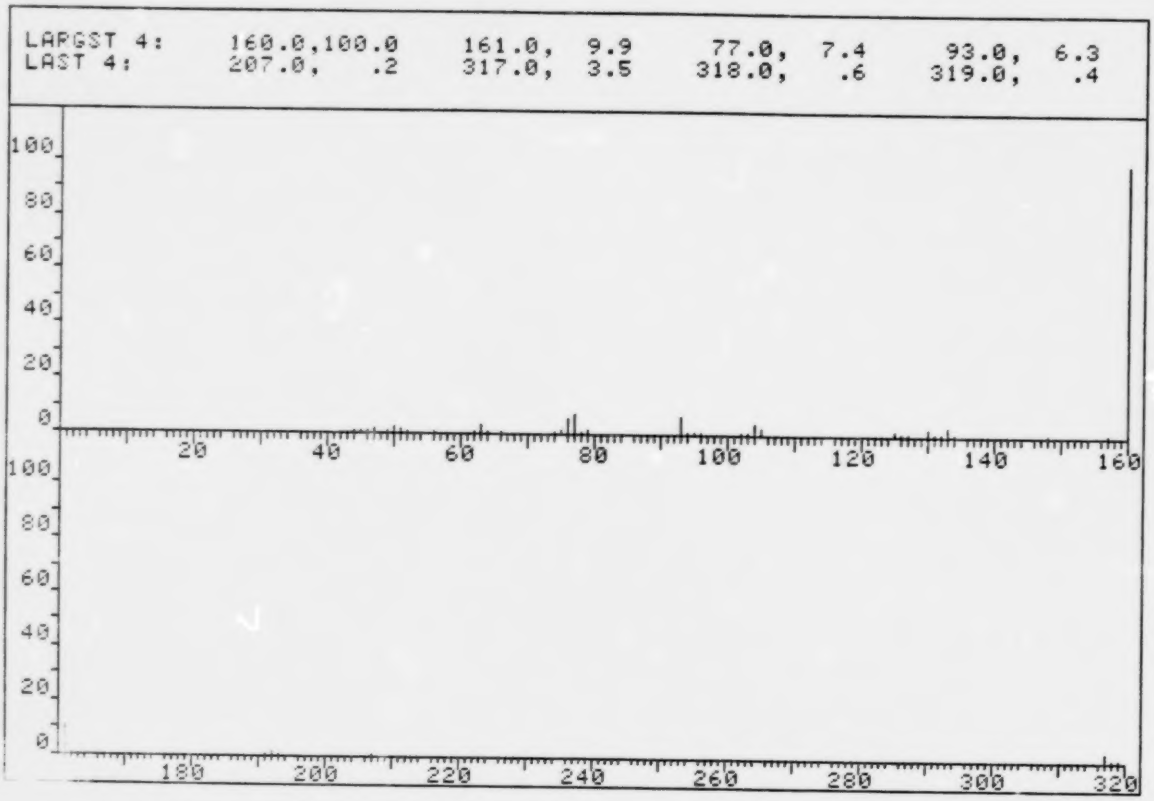


Figure D-44. Calibration curve for the determination of phosphorodithioic acid, S-(((p-chlorophenyl)thio)methyl) O,O-diethyl ester by GC/MS.

Range: 19 - 150 ng
Correlation coefficient: 0.995
Slope: 1.3×10^{-2}
y-Intercept: -4.5×10^{-2}



```
LARGST 4:     160.0,100.0    161.0,  9.9     77.0,  7.4      93.0,  6.3
LAST 4:       207.0,    .2   317.0,  3.5    318.0,   .6     319.0,   .4
```

Figure D-45. Mass spectrum for phosphorodithioic acid, O,O-dimethyl ester, S-ester with N-(mercaptomethyl)phthalimide.

TABLE D-19. KEY IONS AND RELATIVE ABUNDANCES FOR PHOSPHORODITHIOIC ACID, O,O-DIMETHYL ESTER, S-ESTER WITH N-(MERCAPTOMETHYL)PHTHALIMIDE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
45	1.0	74	.9	102	1.1	161	9.9
47	1.8	75	1.3	104	3.9	162	.8
50	2.5	76	5.5	105	2.0	192	1.6
51	1.7	77	7.4	125	1.4	317	3.5
62	.6	78	.8	130	2.0	318	.6
63	3.2	79	1.6	133	3.1		
64	.7	93	6.3	160	100.0		

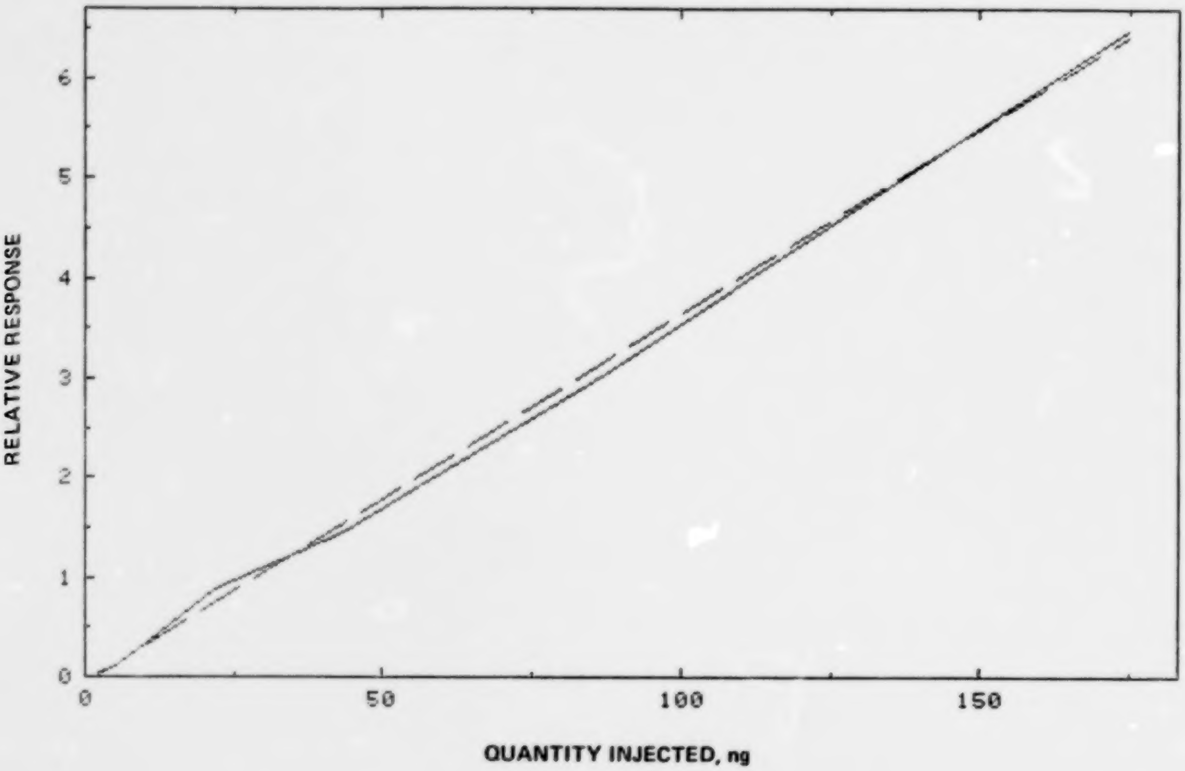


Figure D-46. Calibration curve for the determination of phosphorodithioic acid, O,O-dimethyl ester, S-ester with N-(mercaptomethyl)phthalimide by GC/MS.

Range: 4.4 - 170 ng
Correlation coefficient: 0.999
Slope: 3.7×10^{-2}
y-Intercept: -6.8×10^{-2}

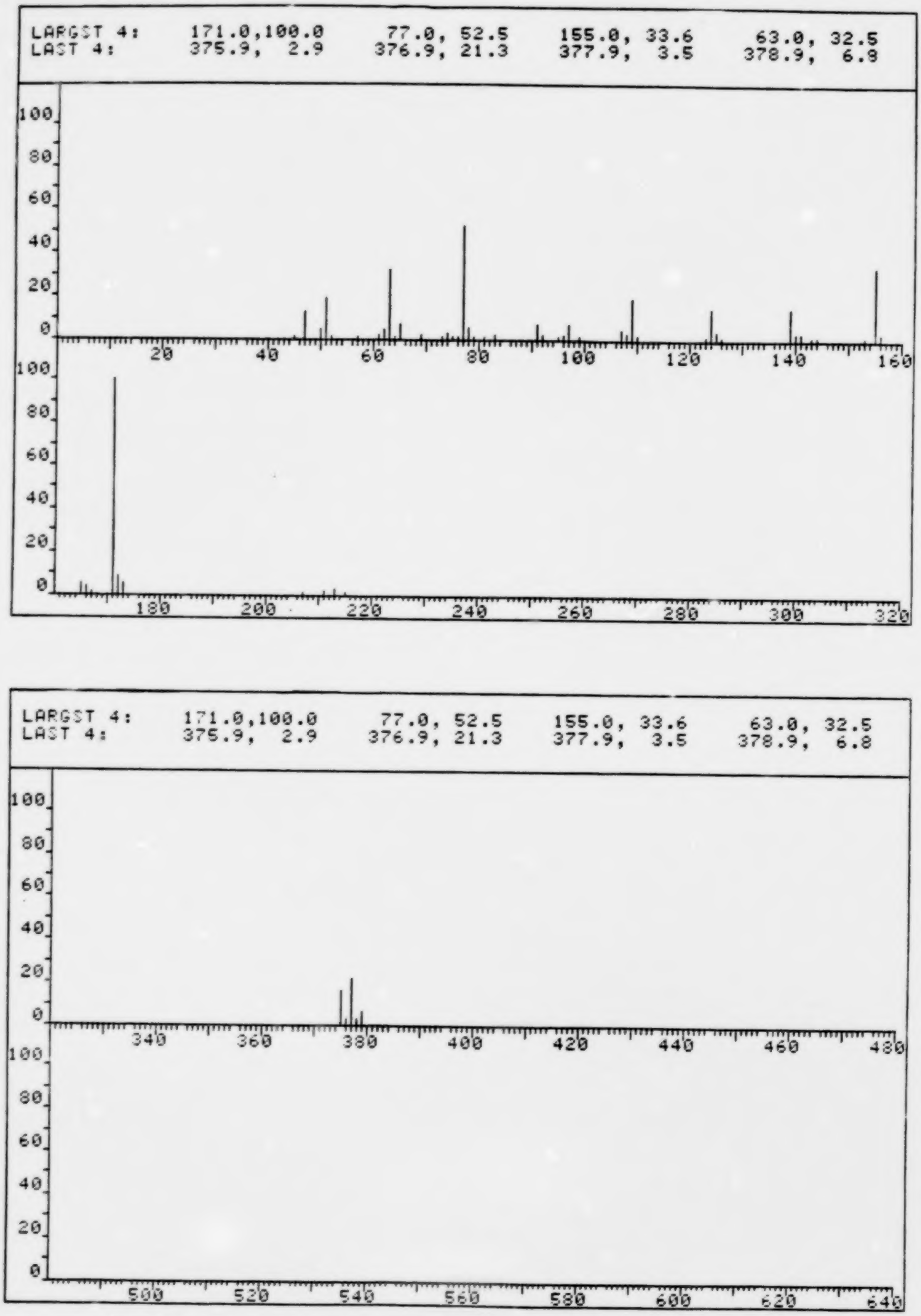


Figure D-47. **Mass spectrum for phosphonothioic acid, phenyl-, O-(4-bromo-2,5-dichloro-phenyl) O-methyl ester.**

TABLE D-20. KEY IONS AND RELATIVE ABUNDANCES FOR PHOSPHONOTHIOIC ACID, PHENYL-, O-(4-BROMO-2,5-DICHLORO-PHENYL) O-METHYL ESTER

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
45	1.7	76	1.9	110	2.2	167	1.3
47	12.7	77	52.5	123	1.3	171	100.0
50	5.3	78	5.8	124	14.4	172	8.6
51	19.7	79	1.3	125	4.3	173	5.1
52	1.3	81	1.4	126	1.3	207	1.6
57	1.3	83	2.1	139	14.7	211	1.7
61	2.2	91	6.9	140	3.6	213	3.1
62	5.1	92	2.2	141	3.4	215	1.6
63	32.5	95	1.4	143	1.4	375	15.8
64	1.3	96	2.1	144	1.3	376	2.9
65	7.0	97	7.1	153	1.7	377	21.3
69	2.1	99	1.9	155	33.6	378	3.5
73	1.8	107	4.8	156	3.1	379	6.8
74	3.8	108	3.1	165	5.3		
75	1.8	109	19.3	166	3.8		

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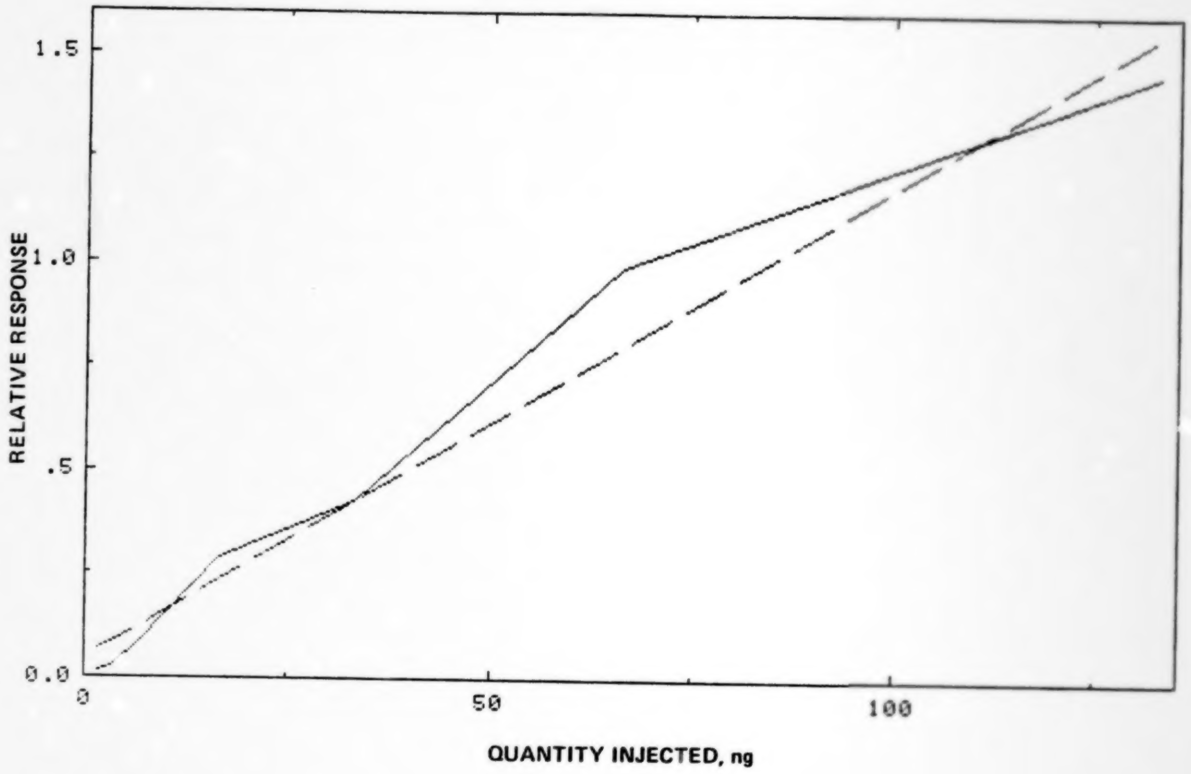
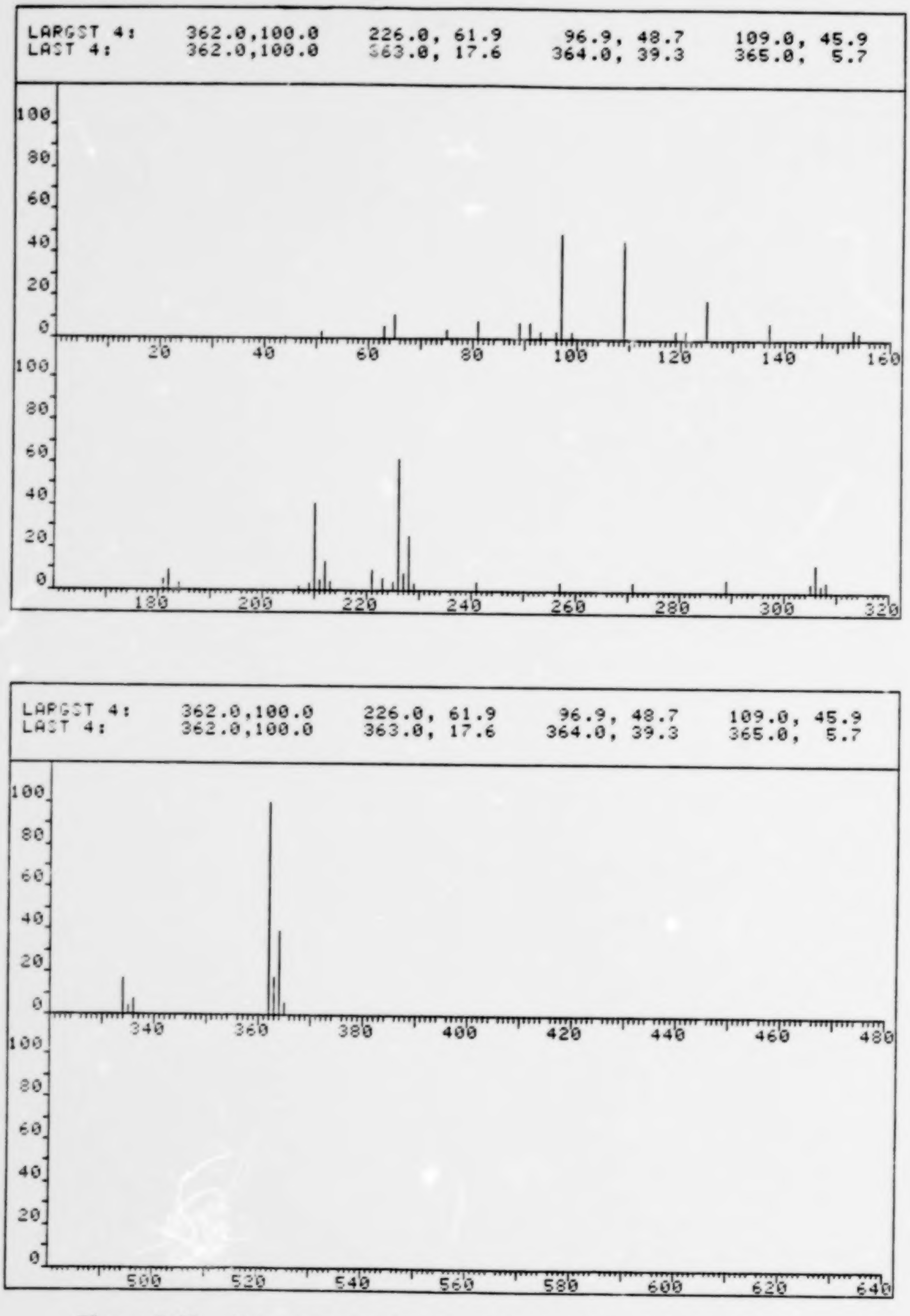


Figure D-48. Calibration curve for the determination of phosphonothioic acid, phenyl-, O-(4-bromo-2,5-dichloro-phenyl) O-methyl ester by GC/MS.

Range: 10 - 130 ng
Correlation coefficient: 0.985
Slope: 1.1×10^{-2}
y-Intercept: 4.7×10^{-2}



```
LARGST 4:     362.0,100.0     226.0, 61.9      96.9, 48.7     109.0, 45.9
LAST 4:       362.0,100.0     363.0, 17.6     364.0, 39.3     365.0,  5.7
```

```
LARGST 4:     362.0,100.0     226.0, 61.9      96.9, 48.7     109.0, 45.9
LAST 4:       362.0,100.0     363.0, 17.6     364.0, 39.3     365.0,  5.7
```

Figure D-49. Mass spectrum for coumarin, 3-chloro-7-hydroxy-4-methyl-, O-ester with O,O-diethyl phosphorothioate.

TABLE D-21. KEY IONS AND RELATIVE ABUNDANCES FOR COUMARIN, 3-CHLORO-7-HYDROXY-4-METHYL-, O-ESTER WITH O,O-DIETHYL PHOSPHOROTHIOATE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	1.3	119	4.1	211	4.7	289	5.7
51	3.1	121	4.1	212	13.8	305	4.1
63	5.7	125	18.6	213	3.8	306	12.3
65	11.6	137	7.9	221	9.7	307	3.5
75	4.1	147	4.1	223	5.0	308	4.7
81	7.9	153	5.0	225	3.8	334	17.0
89	6.9	154	3.5	226	61.9	335	4.1
91	7.5	181	4.7	227	8.2	336	7.2
93	3.8	182	9.7	228	25.5	362	100.0
96	3.1	184	3.1	229	3.5	363	17.6
97	48.7	207	1.6	241	3.8	364	39.3
99	3.8	209	3.1	257	3.8	365	5.7
109	45.9	210	40.9	271	3.8		

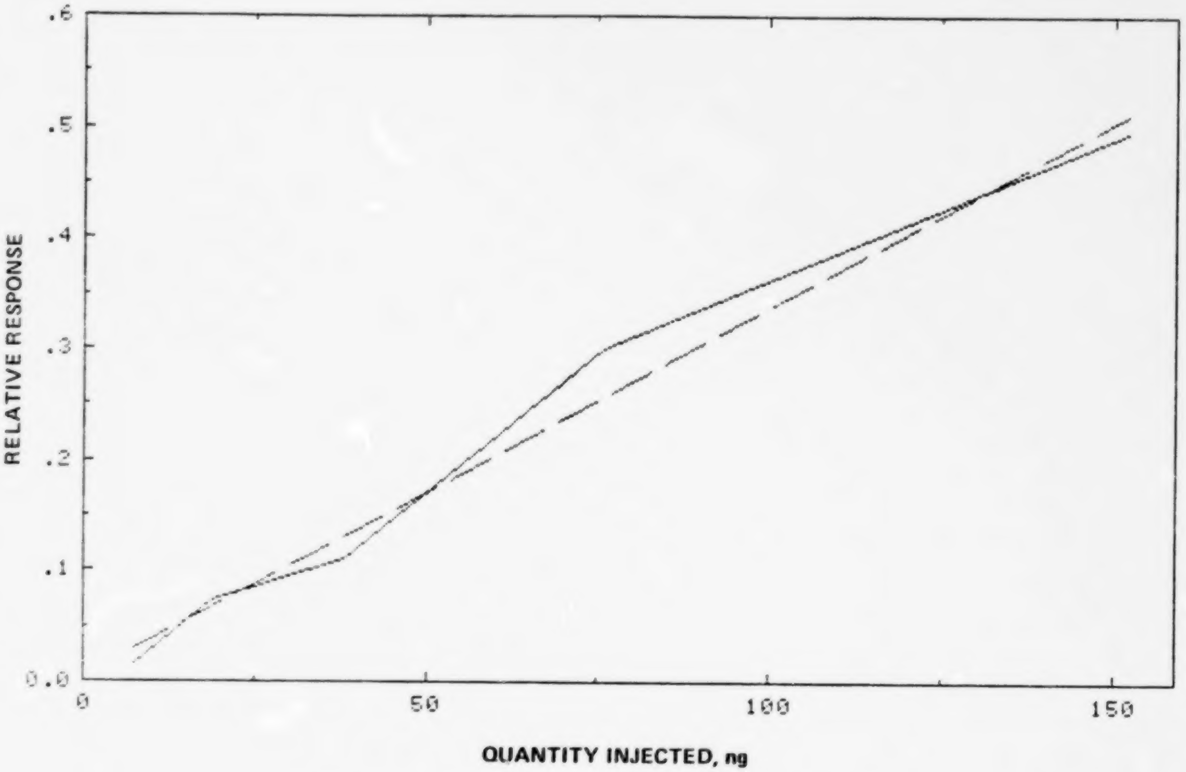


Figure D-50. Calibration curve for the determination of coumarin, 3-chloro-7-hydroxy-4-methyl-, O-ester with O,O-diethyl phosphorothioate by GC/MS.

Range: 10 - 150 ng
Correlation coefficient: 0.992
Slope: 3.3×10^{-3}
y-Intercept: 3.6×10^{-3}

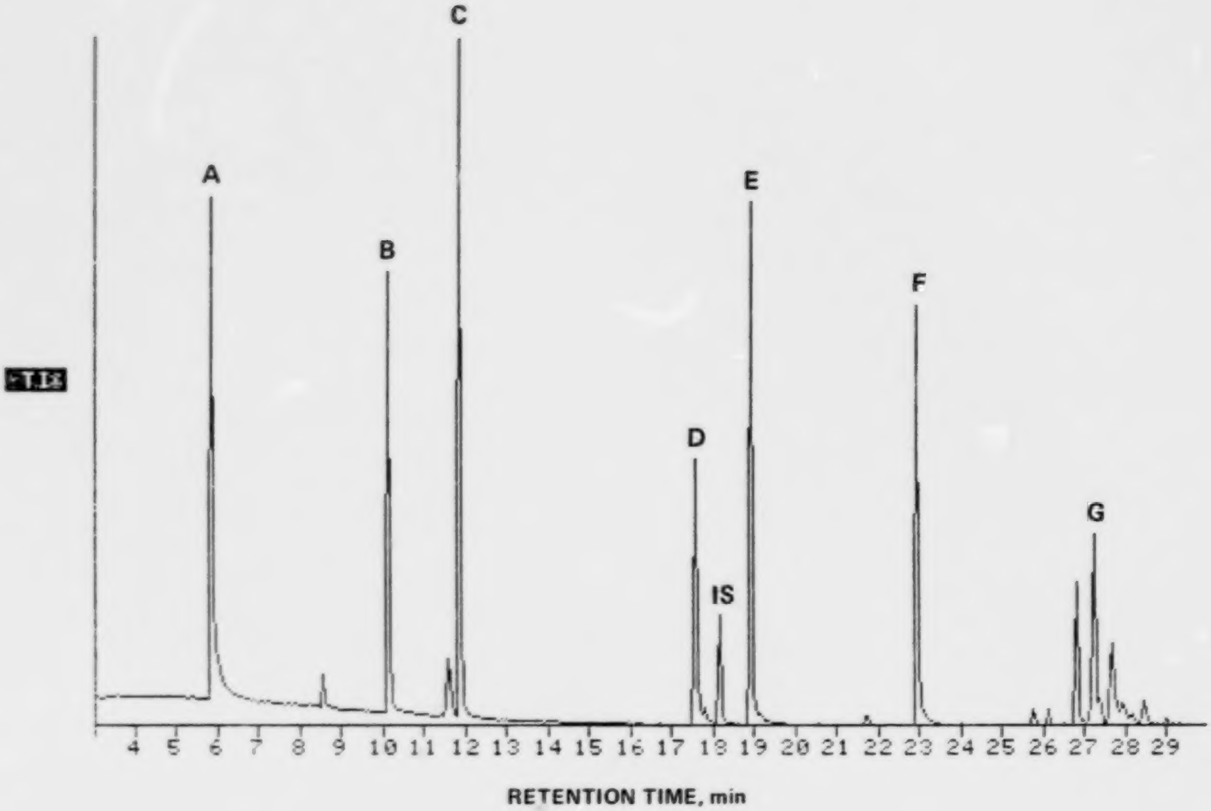
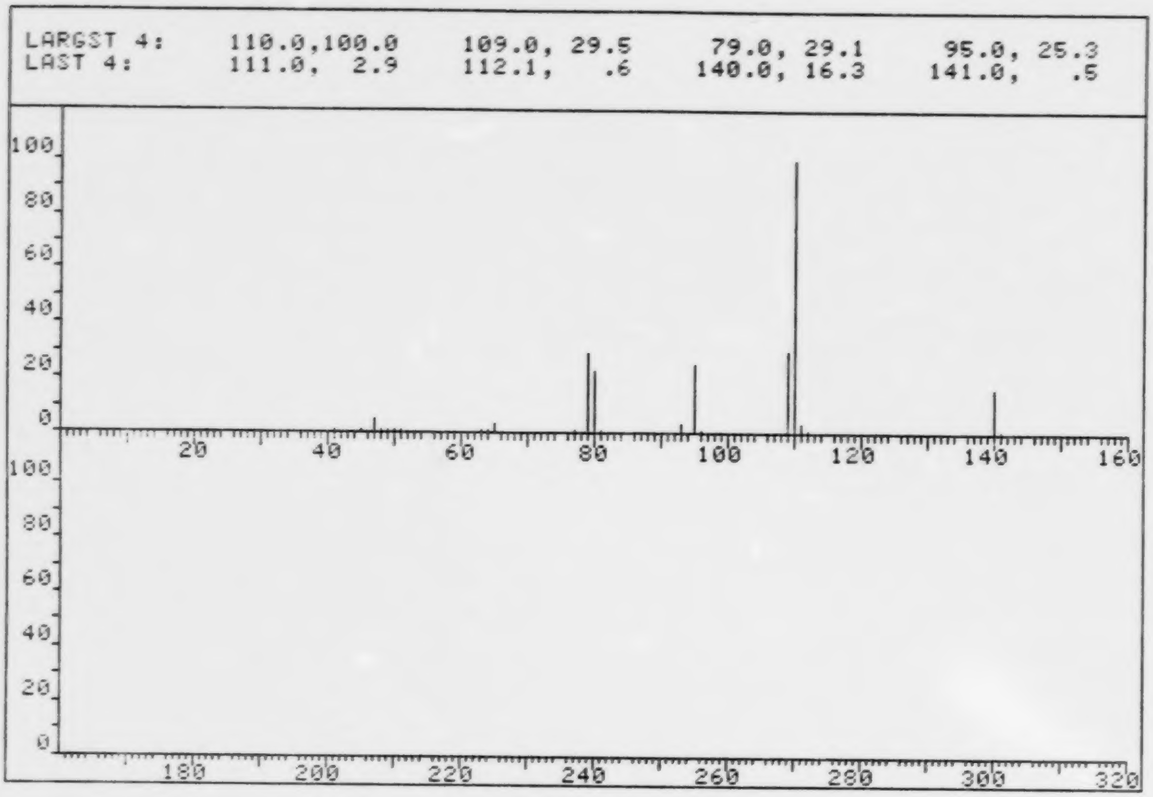


Figure D-51. Chromatogram by GC/MS for

(A) phosphoric acid, trimethyl ester(t_R = 5.9 min),
(B) o-anisidine(t_R = 10.1 min),
(C) aniline, 2,4,5-trimethyl- (t_R = 11.9 min),
(D) o-anisidine, 5-nitro- (t_R = 17.6 min),
(E) biphenyl, 4-nitro(t_R = 18.9 min),
(F) o-toluidine, 4-(o-tolylazo)- (t_R = 23.0 min), and
(G) phosphoric acid, tri-o-tolyl ester(t_R = 27.3 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal



```
LARGST 4:     110.0,100.0     109.0, 29.5      79.0, 29.1      95.0, 25.3
LAST 4:       111.0,  2.9     112.1,   .6     140.0, 16.3     141.0,   .5
```

Figure D-52. Mass spectrum for phosphoric acid, trimethyl ester.

TABLE D-22. KEY IONS AND RELATIVE ABUNDANCES FOR PHOSPHORIC ACID, TRIMETHYL ESTER

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
45	.6	65	3.6	93	2.8	111	2.9
47	4.7	77	1.0	95	25.3	112	.6
48	.7	79	29.1	96	.7	140	16.3
63	.8	80	23.0	109	29.5	141	.5
64	.5	81	.9	110	100.0		

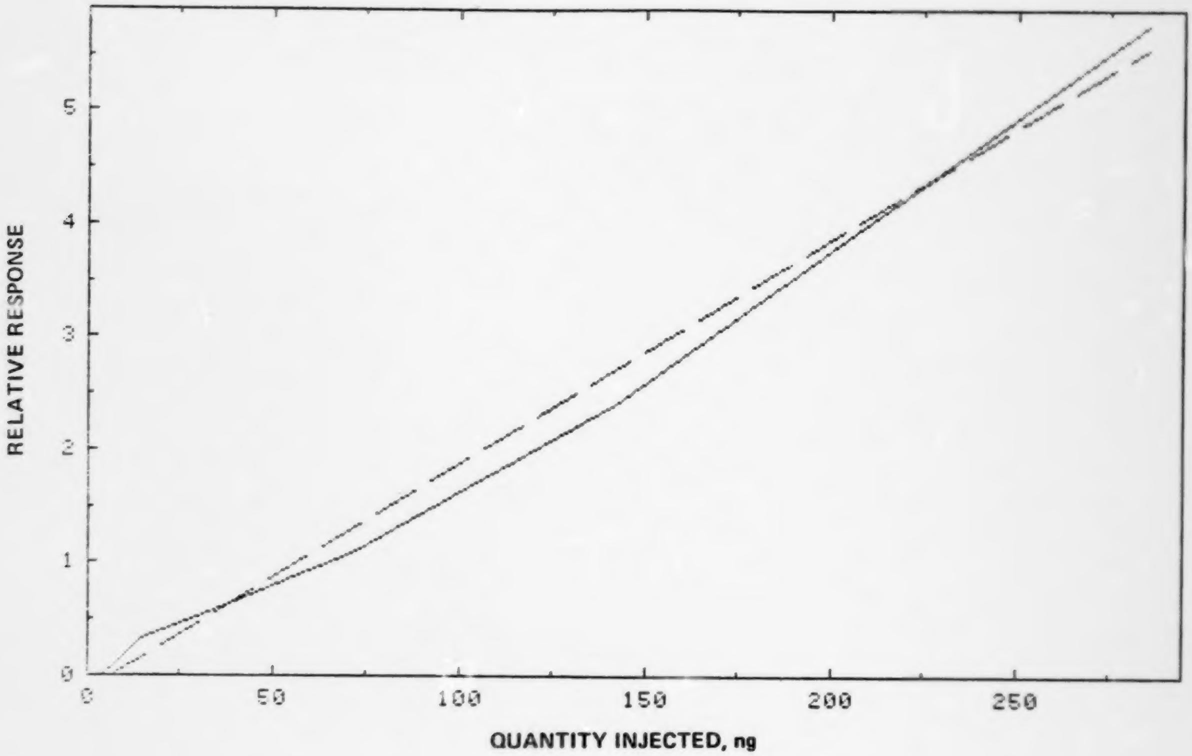
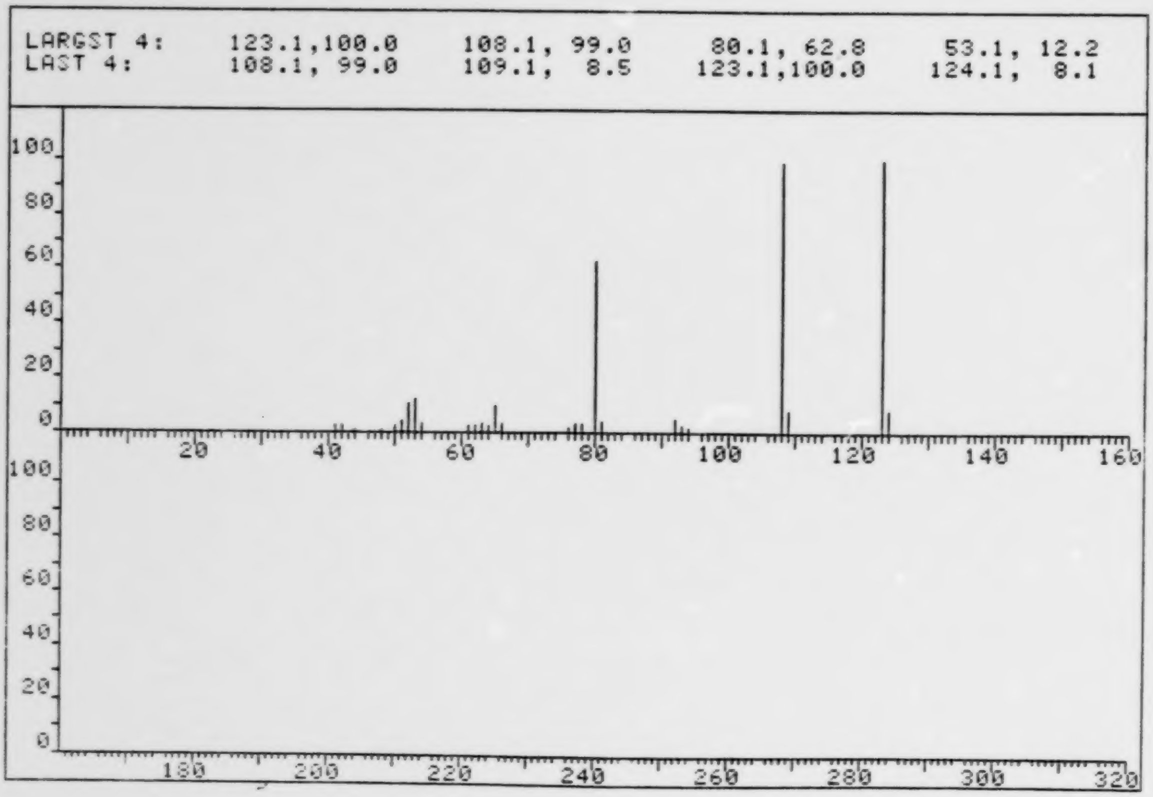


Figure D-53. Calibration curve for the determination of phosphoric acid, trimethyl ester by GC/MS.

Range: 90 - 280 ng
Correlation coefficient: 0.996
Slope: 2.0×10^{-2}
y-Intercept: -1.3×10^{-1}



```
LARGST 4:     123.1,100.0     108.1, 99.0      80.1, 62.8      53.1, 12.2
LAST 4:       108.1, 99.0     109.1,  8.5     123.1,100.0     124.1,  8.1
```

Figure D-54. Mass spectrum for o-anisidine.

TABLE D-23. KEY IONS AND RELATIVE ABUNDANCES FOR o-ANISIDINE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	2.4	54	3.7	76	2.0	94	2.0
42	2.2	61	2.6	77	3.3	108	99.0
48	.8	62	2.4	78	2.8	109	8.5
50	2.2	63	2.8	80	62.8	123	100.0
51	4.3	64	2.6	81	4.5	124	8.1
52	10.4	65	10.2	92	4.7		
53	12.2	66	3.0	93	2.6		

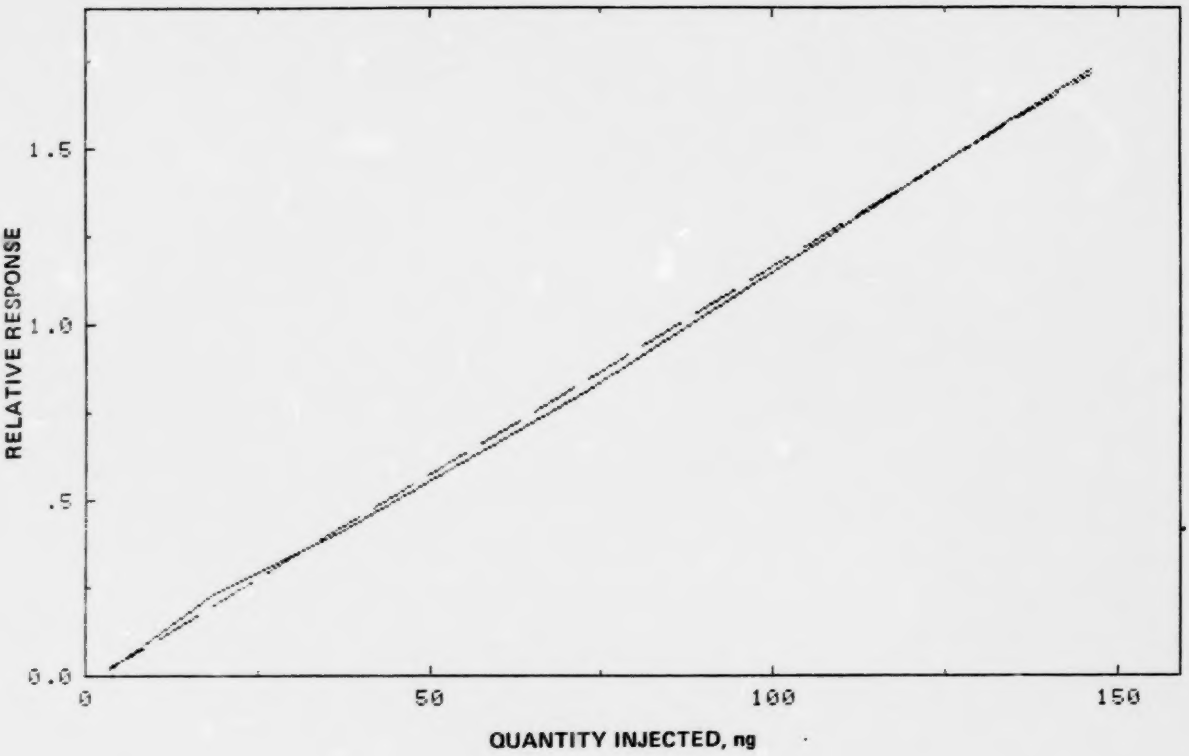
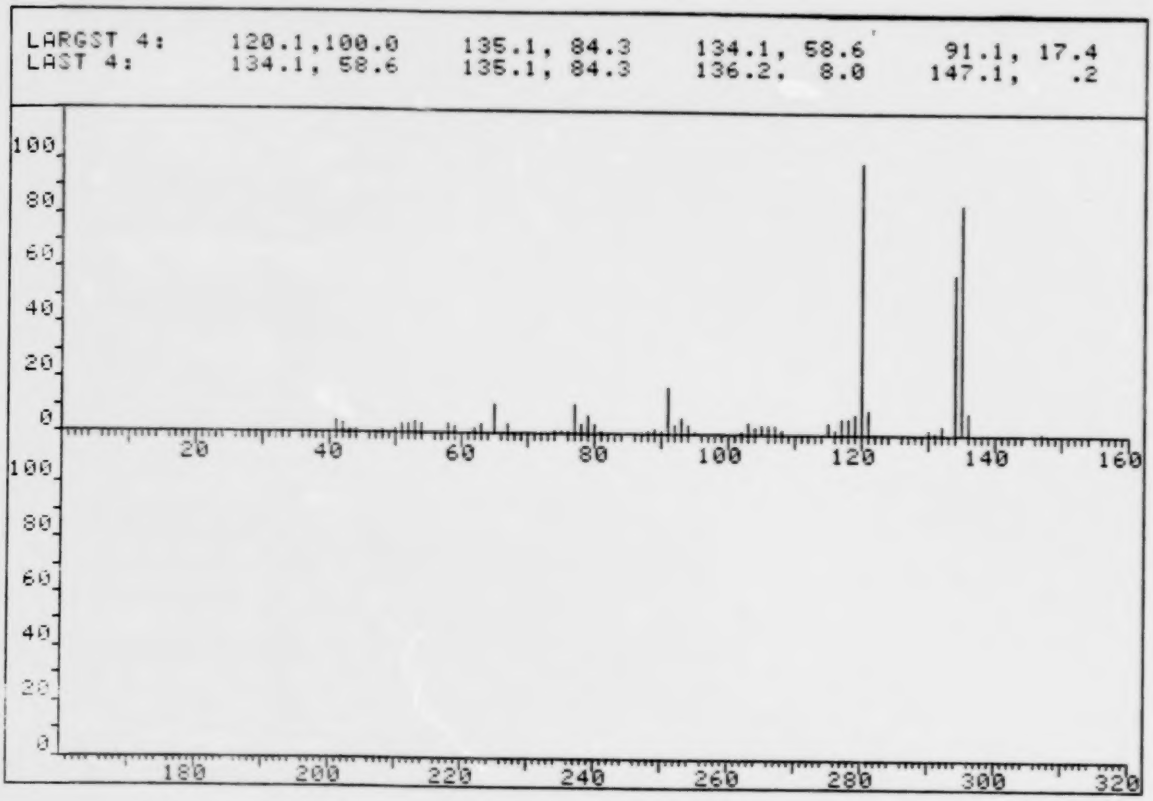


Figure D-55. Calibration curve for the determination of *o*-anisidine by GC/MS.

Range: 3.7 - 150 ng
Correlation coefficient: 0.999
Slope: 1.2×10^{-2}
y-Intercept: -2.1×10^{-2}



```
LARGST 4:     120.1,100.0     135.1, 84.3     134.1, 58.6      91.1, 17.4
LAST 4:       134.1, 58.6     135.1, 84.3     136.2,  8.0     147.1,    .2
```

Figure D-56. Mass spectrum for aniline, 2,4,5-trimethyl-.

TABLE D-24. KEY IONS AND RELATIVE ABUNDANCES FOR ANILINE, 2,4,5-TRIMETHYL-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	4.2	63	3.6	91	17.4	116	1.9
42	3.5	65	10.5	92	3.4	117	5.5
43	.8	67	3.7	93	5.7	118	5.7
44	.6	74	.9	94	3.2	119	6.9
48	.8	75	1.3	95	.9	120	100.0
50	1.4	76	1.3	102	1.1	121	9.3
51	3.6	77	10.3	103	4.3	130	1.4
52	3.8	78	3.7	104	2.4	132	3.9
53	4.3	79	6.6	105	3.1	134	58.6
54	3.0	80	3.4	106	3.3	135	84.3
58	3.4	81	.8	107	3.0	136	8.0
59	2.3	89	2.0	108	1.9		
62	1.8	90	1.3	115	4.3		

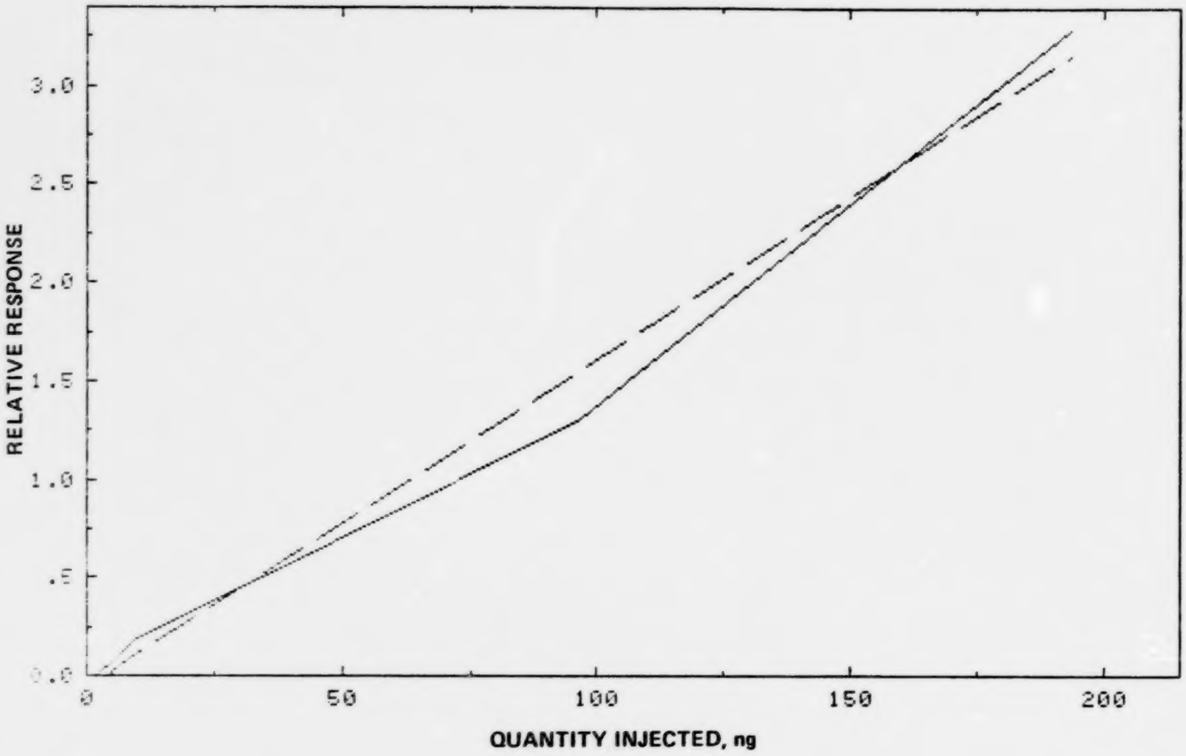
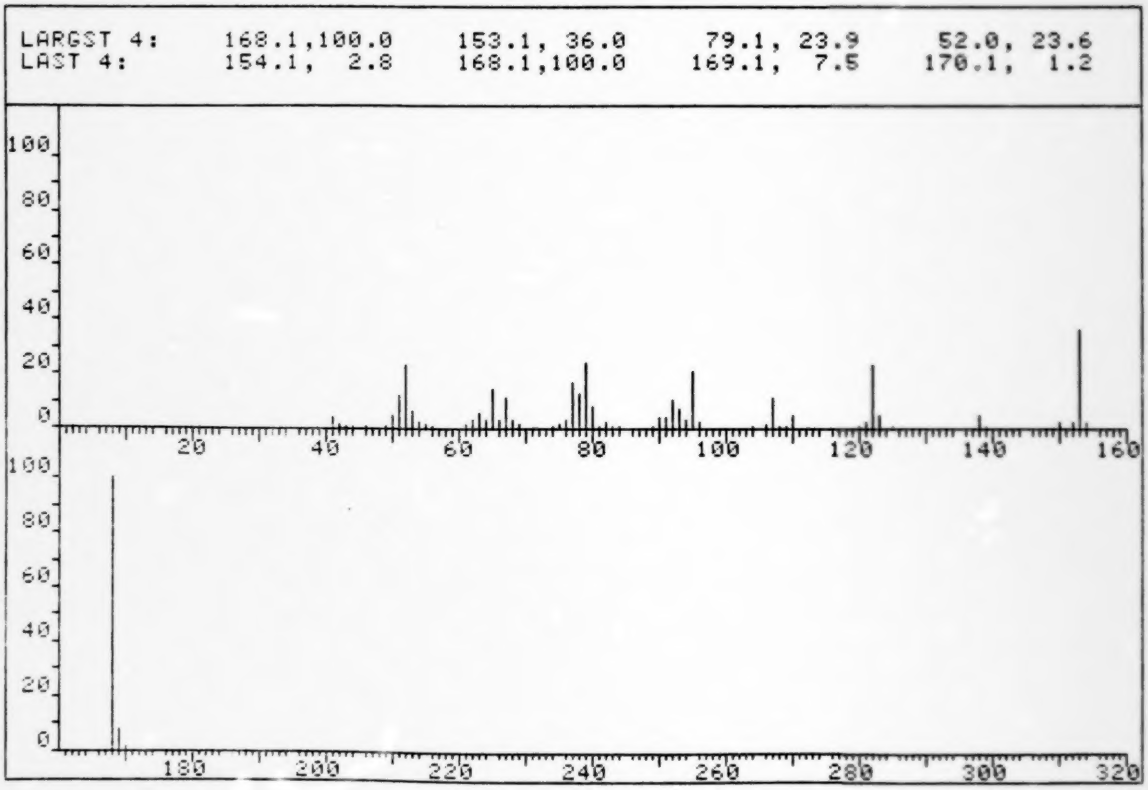


Figure D-57. Calibration curve for the determination of aniline, 2,4,5-trimethyl- by GC/MS.

Range: 10 - 190 ng
Correlation coefficient: 0.995
Slope: 1.7×10^{-2}
y-Intercept: -5.3×10^{-2}



```
LARGST 4:    168.1,100.0    153.1, 36.0     79.1, 23.9     52.0, 23.6
LAST 4:      154.1,   2.8   168.1,100.0    169.1,   7.5   170.1,   1.2
```

Figure D-58. Mass spectrum for o-anisidine, 5-nitro-.

TABLE D-25. KEY IONS AND RELATIVE ABUNDANCES FOR *o*-ANISIDINE, 5-NITRO-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	4.1	63	5.6	82	2.5	110	5.4
42	1.8	64	3.5	83	.6	120	.9
43	.6	65	14.9	89	1.1	121	2.5
44	1.0	66	3.1	90	4.5	122	23.4
46	1.1	67	11.5	91	3.9	123	5.0
49	.6	68	3.2	92	10.3	125	.9
50	4.7	69	1.4	93	7.2	138	4.8
51	11.8	74	1.3	94	3.1	139	.8
52	23.6	75	1.3	95	20.7	150	2.3
53	6.4	76	3.1	96	2.4	152	2.5
54	2.3	77	16.7	104	.6	153	36.0
55	1.3	78	13.0	106	2.0	154	2.8
56	.6	79	23.9	107	11.3	168	100.0
61	1.4	80	8.6	108	1.2	169	7.5
62	3.3	81	.9	109	.6	170	1.2

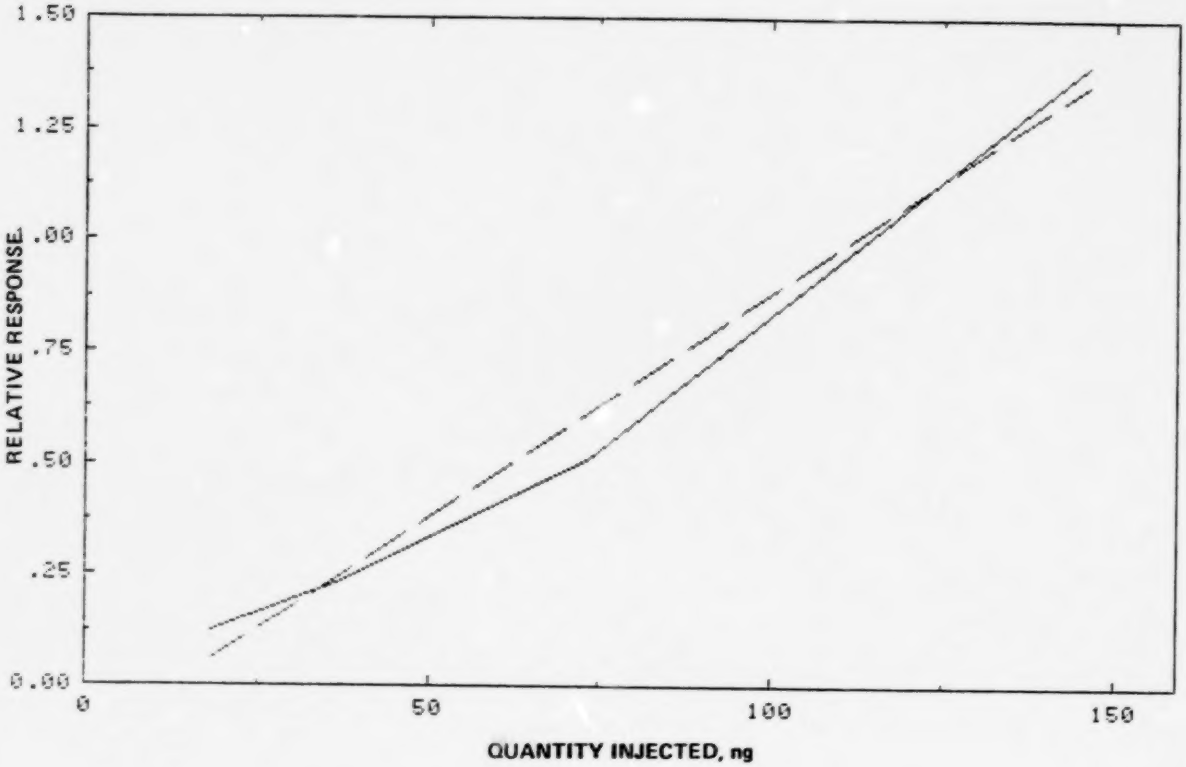
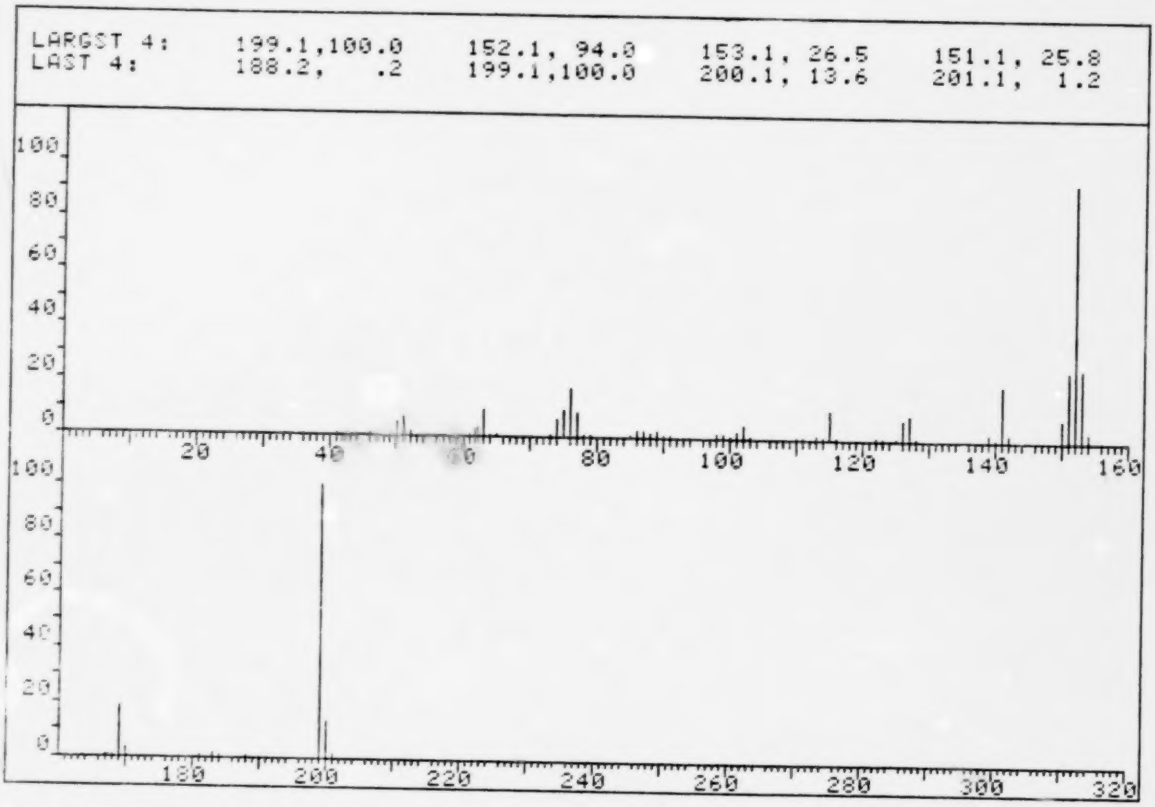


Figure D-59. Calibration curve for the determination of o-anisidine, 5-nitro- by GC/MS.

Range: 25 - 150 ng
Correlation coefficient: 0.992
Slope: 1.0×10^{-2}
y-Intercept: -1.3×10^{-1}



```
LARGST 4:     199.1,100.0     152.1, 94.0     153.1, 26.5     151.1, 25.8
LAST 4:       188.2,    .2    199.1,100.0     200.1, 13.6     201.1,  1.2
```

Figure D-60. Mass spectrum for biphenyl, 4-nitro.

TABLE D-26. KEY IONS AND RELATIVE ABUNDANCES FOR BIPHENYL, 4-NITRO

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
46	1.0	78	1.2	111	.7	142	2.4
50	5.4	85	.6	113	1.4	150	7.8
51	7.3	86	2.2	114	.8	151	25.8
52	1.9	87	2.7	115	10.7	152	94.0
61	1.0	88	1.3	116	1.1	153	26.5
62	3.1	89	2.5	122	.7	154	2.8
63	10.1	91	.6	125	1.1	168	.9
64	1.1	98	2.0	126	7.4	169	18.5
65	1.1	99	1.5	127	8.9	170	2.9
74	6.2	100	1.0	128	1.3	183	1.1
75	9.5	101	2.6	139	2.2	199	100.0
76	18.1	102	5.1	140	1.2	200	13.6
77	9.1	103	1.0	141	20.2	201	1.2

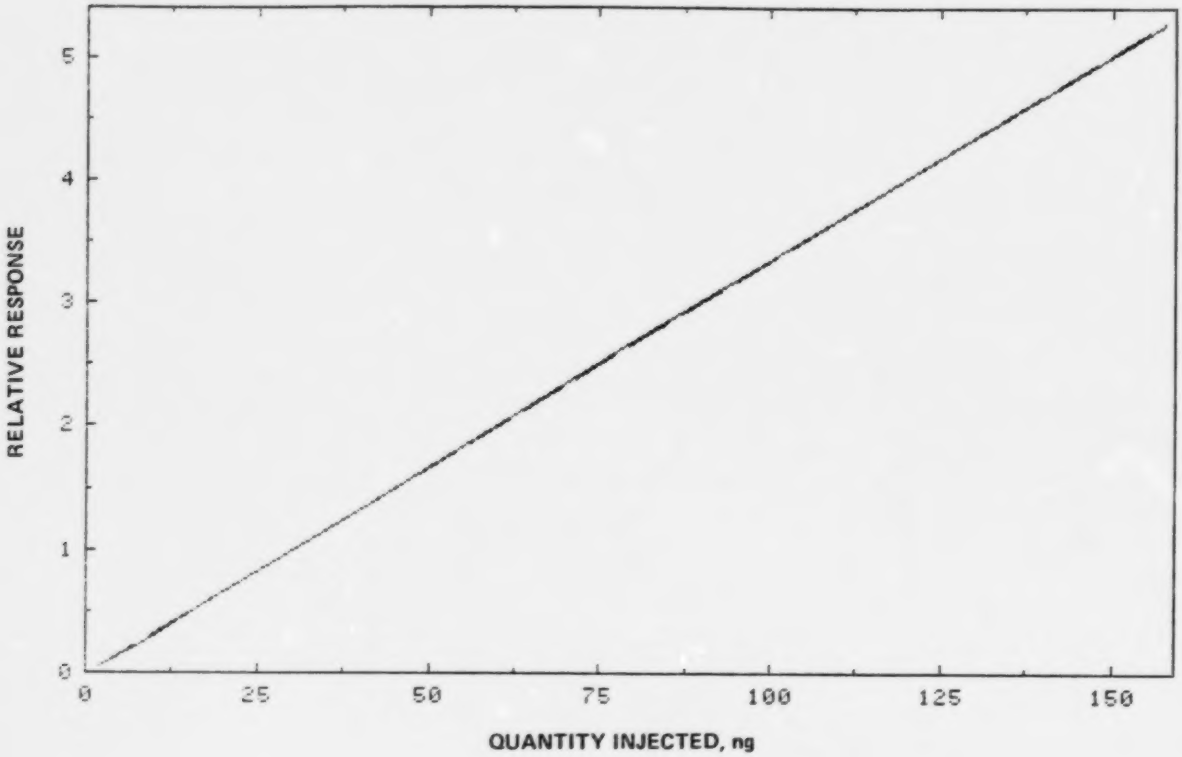


Figure D-61. Calibration curve for the determination of biphenyl, 4-nitro by GC/MS.

Range: 2.0 - 160 ng
Correlation coefficient: 0.999
Slope: 1.6 x 10^{-2}
y-Intercept: -2.1 x 10^{-2}

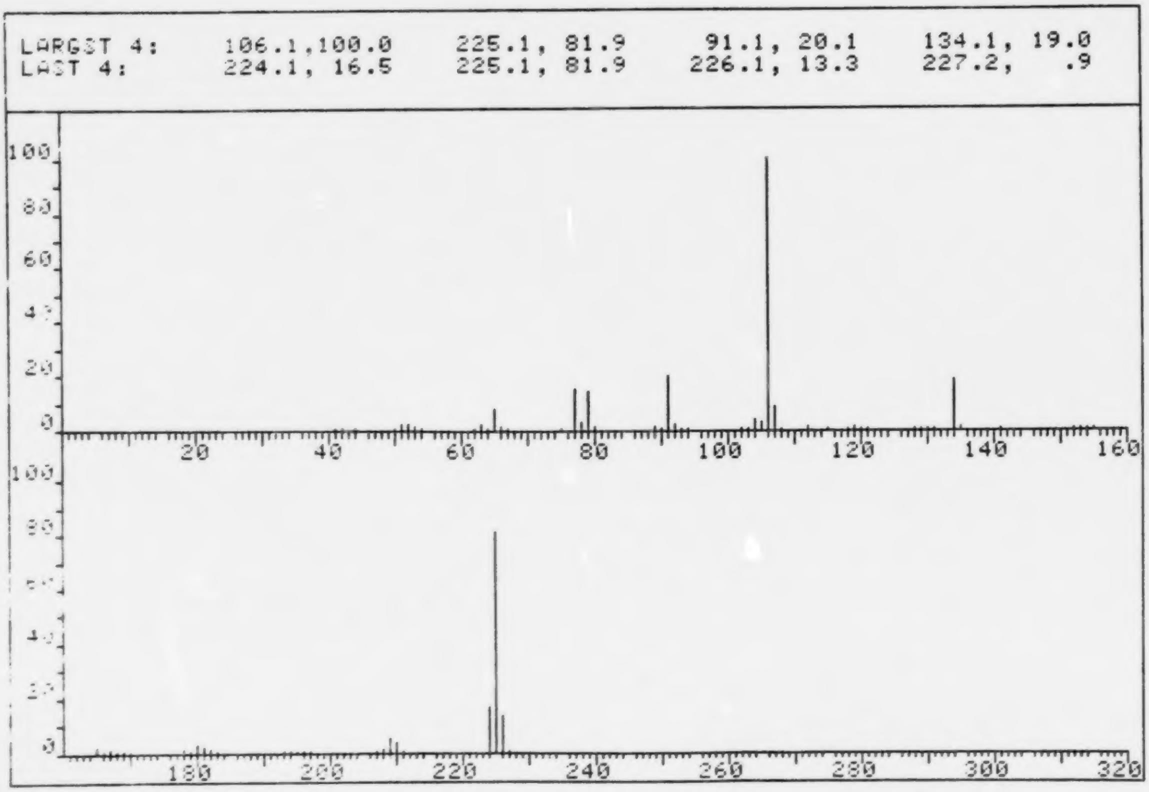


Figure D-62. Mass spectrum for o-toluidine, 4-(o-tolylazo)-.

TABLE D-27. KEY IONS AND RELATIVE ABUNDANCES FOR *o*-TOLUIDINE, 4-(*o*-TOYLYAZO)-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	1.2	80	1.8	120	.6	180	3.3
42	.5	89	2.0	121	1.0	181	2.0
50	.9	90	1.0	128	.5	182	1.5
51	2.5	91	20.1	130	.5	194	.7
52	2.0	92	2.4	134	19.0	196	.9
53	1.9	93	.7	135	1.9	197	.5
54	1.0	94	.6	141	.5	207	.9
63	2.3	104	4.3	152	.9	208	1.2
64	.9	105	2.9	153	.7	209	5.5
65	8.4	106	100.0	165	1.7	210	3.7
66	1.7	107	9.2	166	.8	224	16.5
67	.7	108	.5	167	1.4	225	81.8
77	15.4	112	1.4	168	.6	226	13.3
78	3.8	115	.6	178	1.1	227	.9
79	14.8	119	1.6	179	.7		

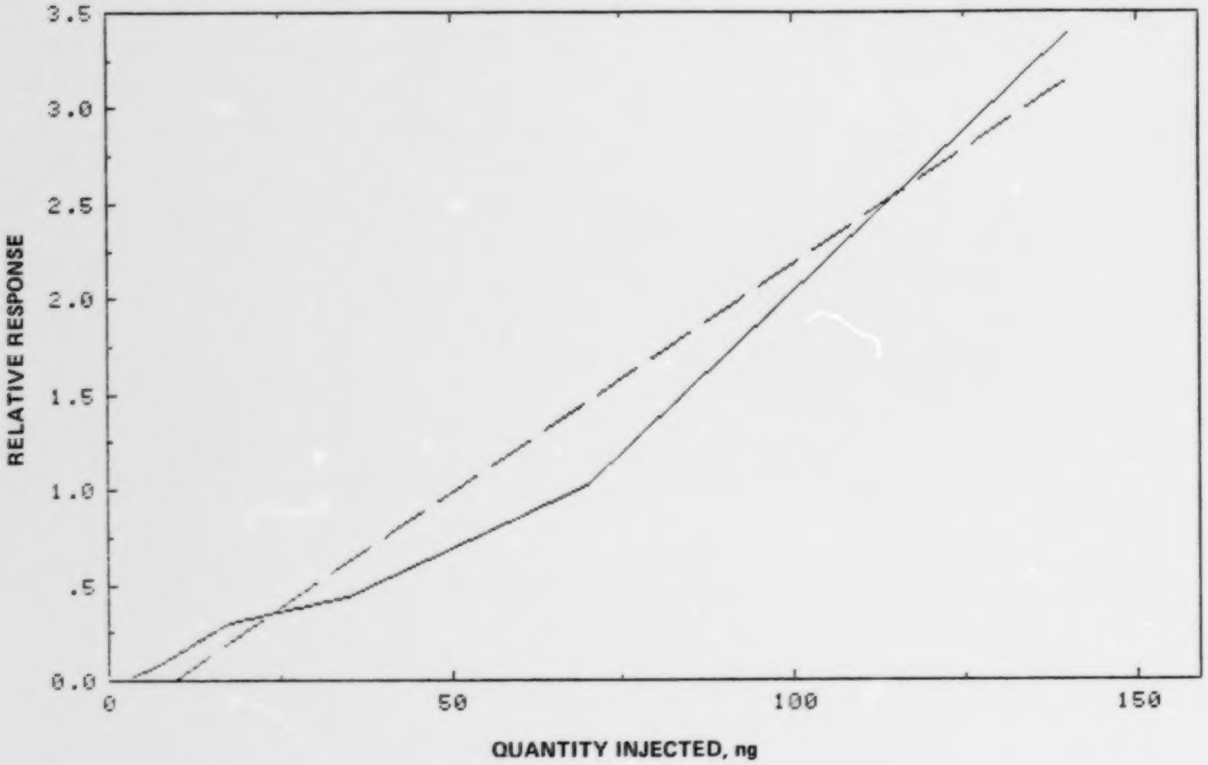
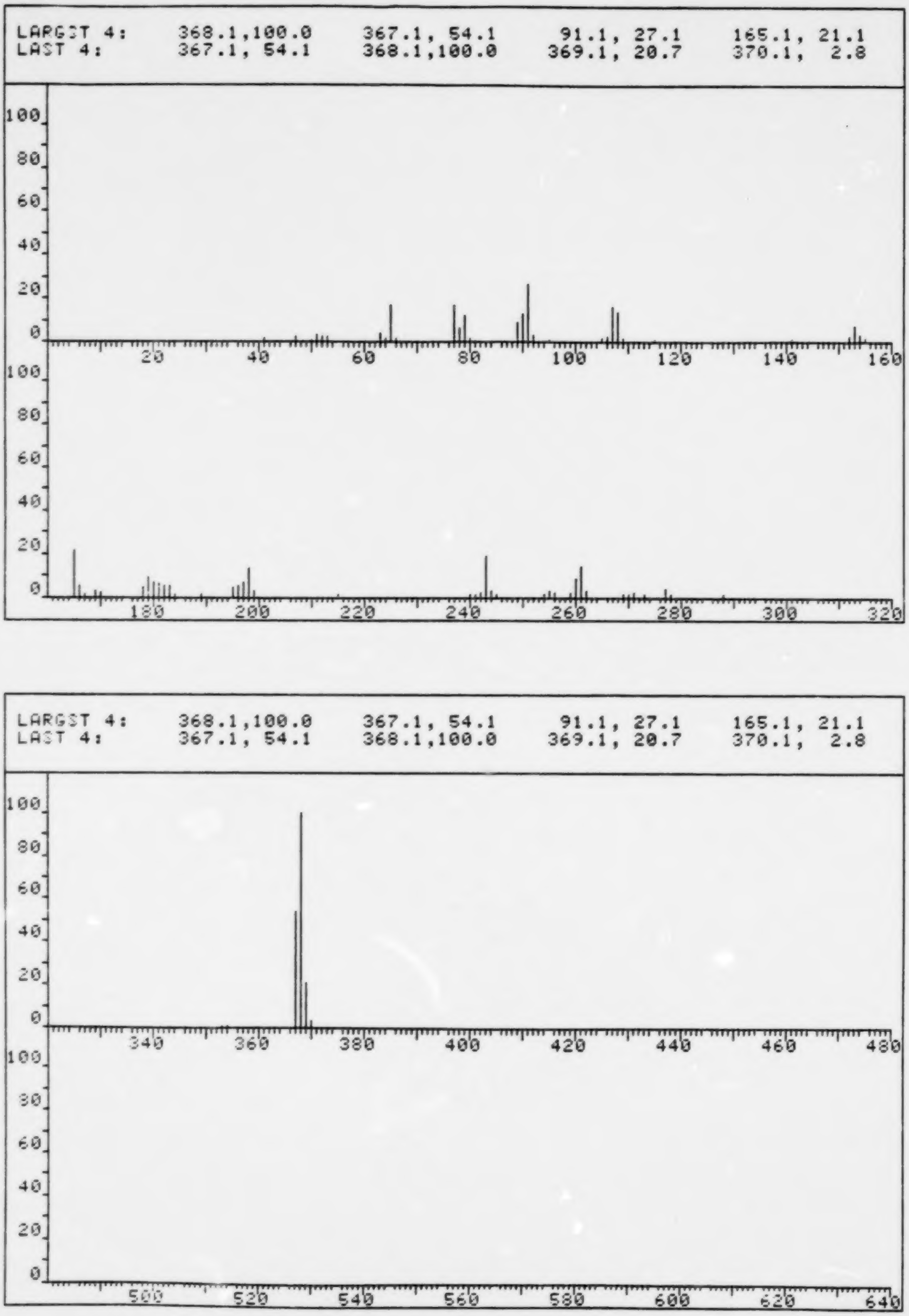


Figure D-63. Calibration curve for the determination of o-toluidine, 4-(o-tolylazo)- by GC/MS.

Range: 25 - 140 ng
Correlation coefficient: 0.980
Slope: 2.4×10^{-2}
y-Intercept: -2.2×10^{-1}



```
LARGST 4:   368.1,100.0   367.1, 54.1    91.1, 27.1   165.1, 21.1
LAST 4:     367.1, 54.1   368.1,100.0   369.1, 20.7   370.1,  2.8
```

```
LARGST 4:   368.1,100.0   367.1, 54.1    91.1, 27.1   165.1, 21.1
LAST 4:     367.1, 54.1   368.1,100.0   369.1, 20.7   370.1,  2.8
```

Figure D-64. Mass spectrum for phosphoric acid, tri-o-tolyl ester.

TABLE D-28. KEY IONS AND RELATIVE ABUNDANCES FOR PHOSPHORIC ACID, TRI-o-TOLYL ESTER

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	1.7	105	1.7	181	5.9	256	2.2
47	2.6	106	2.5	182	5.4	259	2.3
50	1.2	107	15.8	183	5.4	260	8.5
51	3.7	108	13.7	184	1.4	261	14.5
52	2.3	109	1.5	189	1.3	262	3.1
53	2.7	115	1.1	195	4.5	269	1.4
63	4.1	141	1.1	196	5.7	270	1.3
64	1.8	152	2.1	197	6.9	271	1.9
65	17.4	153	7.2	198	13.4	273	1.1
66	1.7	154	3.8	199	2.7	277	3.8
77	17.0	155	1.5	215	1.3	278	1.1
78	6.7	165	21.1	240	1.2	288	1.1
79	12.1	166	5.2	241	1.2	367	54.1
80	1.3	167	1.3	242	2.2	368	100.0
89	9.2	169	2.5	243	19.2	369	20.7
90	12.6	170	2.1	244	3.3	370	2.8
91	27.1	178	4.8	245	1.6		
92	2.9	179	9.2	254	1.4		
95	1.1	180	6.7	255	3.3		

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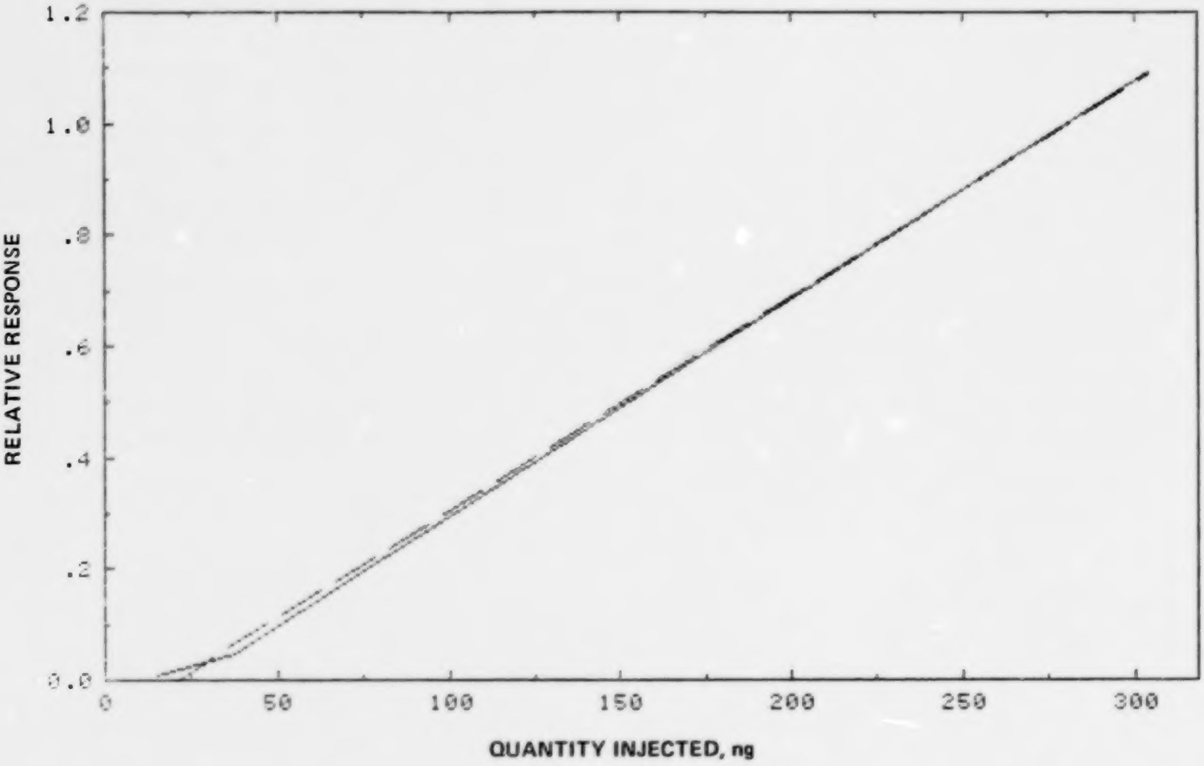


Figure D-65. Calibration curve for the determination of phosphoric acid, tri-o-tolyl ester by GC/MS.

Range: 30 - 300 ng
Correlation coefficient: 0.999
Slope: 3.8 x 10^{-3}
y-Intercept: -7.9 x 10^{-2}

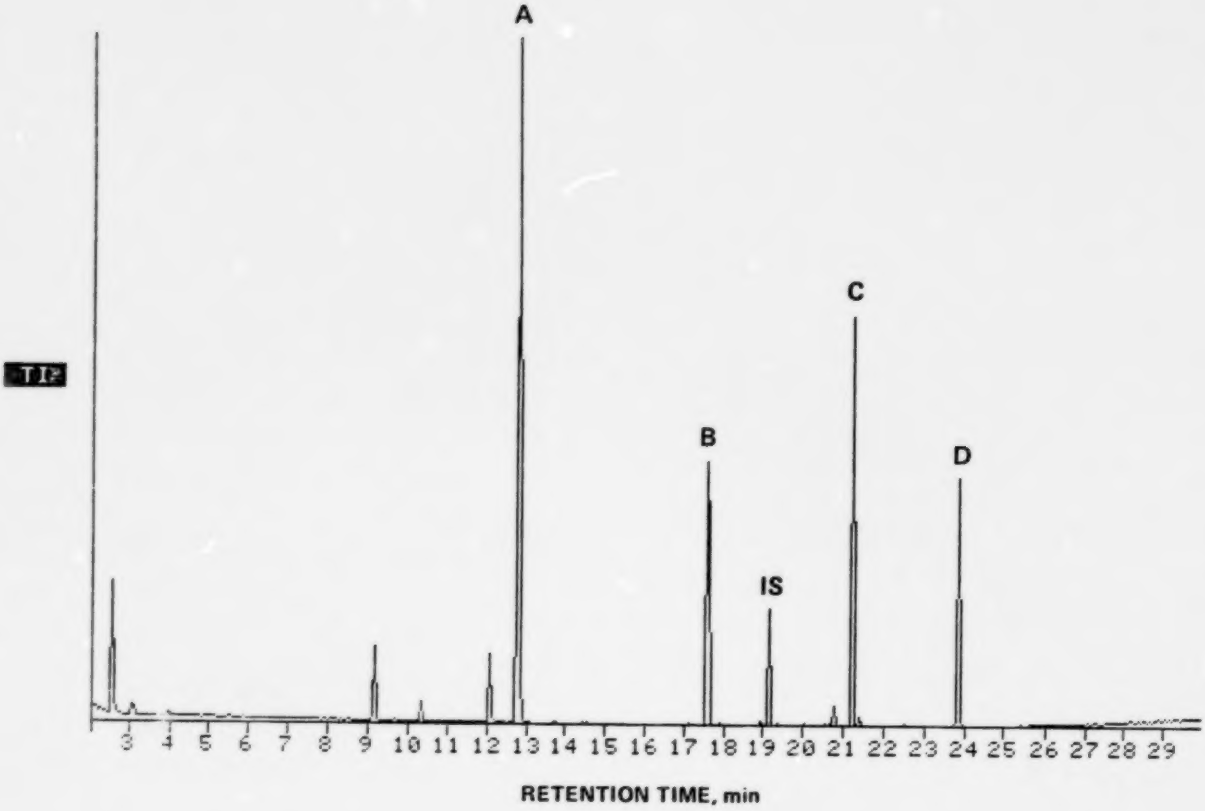
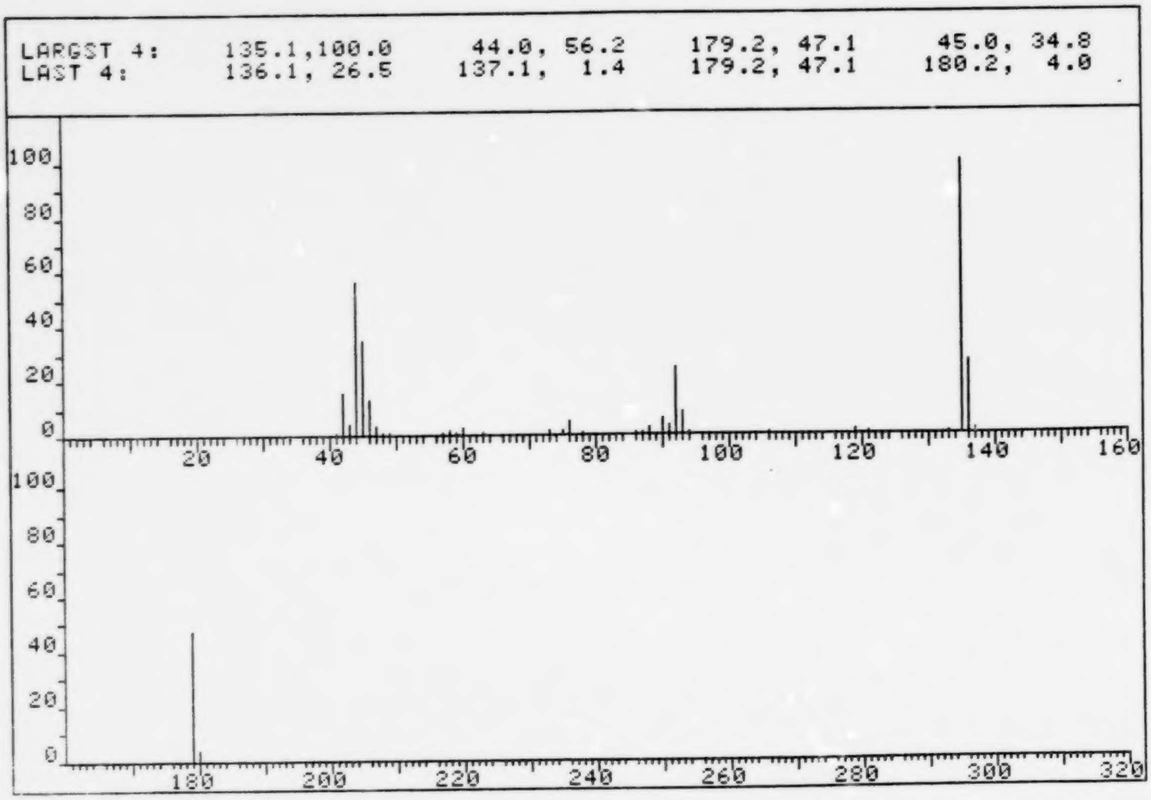


Figure D-66. Chromatogram by GC/MS for

(A) phosphoric triamide, hexamethyl- (t_R = 12.8 min),
(B) phosphoric acid, 1,2-dibromo-2,2-dichloroethyl dimethyl ester(t_R = 17.6 min),
(C) phosphorothioic acid, O,O-dimethyl-, O-(4-(methylthio)-m-tolyl) ester(t_R = 21.2 min), and
(D) phosphorothioic acid, O,O-diethyl O-(p-(methylsulfinyl) phenyl) ester(t_R = 23.9 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal



```
LARGST 4:    135.1,100.0     44.0, 56.2    179.2, 47.1     45.0, 34.8
LAST 4:      136.1, 26.5    137.1,  1.4    179.2, 47.1    180.2,  4.0
```

Figure D-67. Mass spectrum for phosphoric triamide, hexamethyl-.

TABLE D-29. KEY IONS AND RELATIVE ABUNDANCES FOR PHOSPHORIC TRIAMIDE, HEXAMETHYL-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	.7	49	1.1	78	1.2	119	1.8
42	15.6	58	1.9	88	2.3	121	1.2
43	3.9	60	2.7	90	5.4	135	100.0
44	56.2	63	.8	91	3.9	136	26.4
45	34.8	73	1.6	92	24.5	137	1.4
46	12.5	75	1.3	93	7.8	179	47.1
47	3.0	76	5.0	106	.7	180	4.0

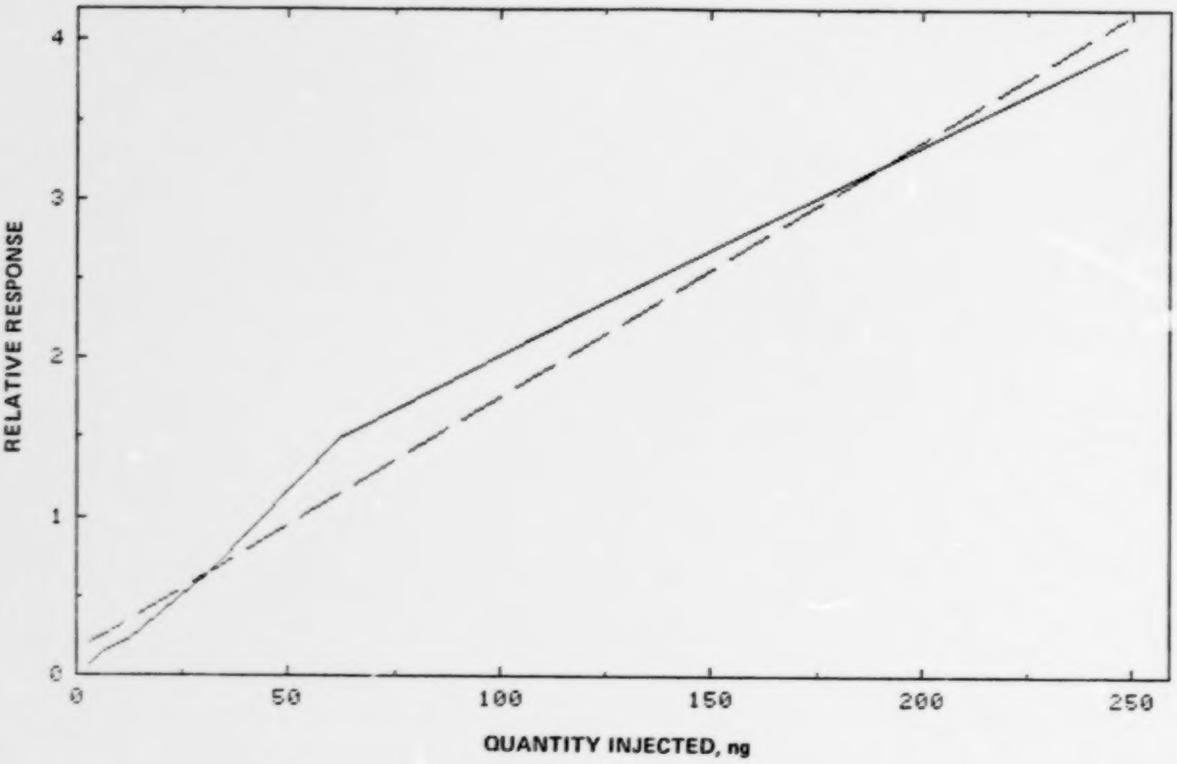
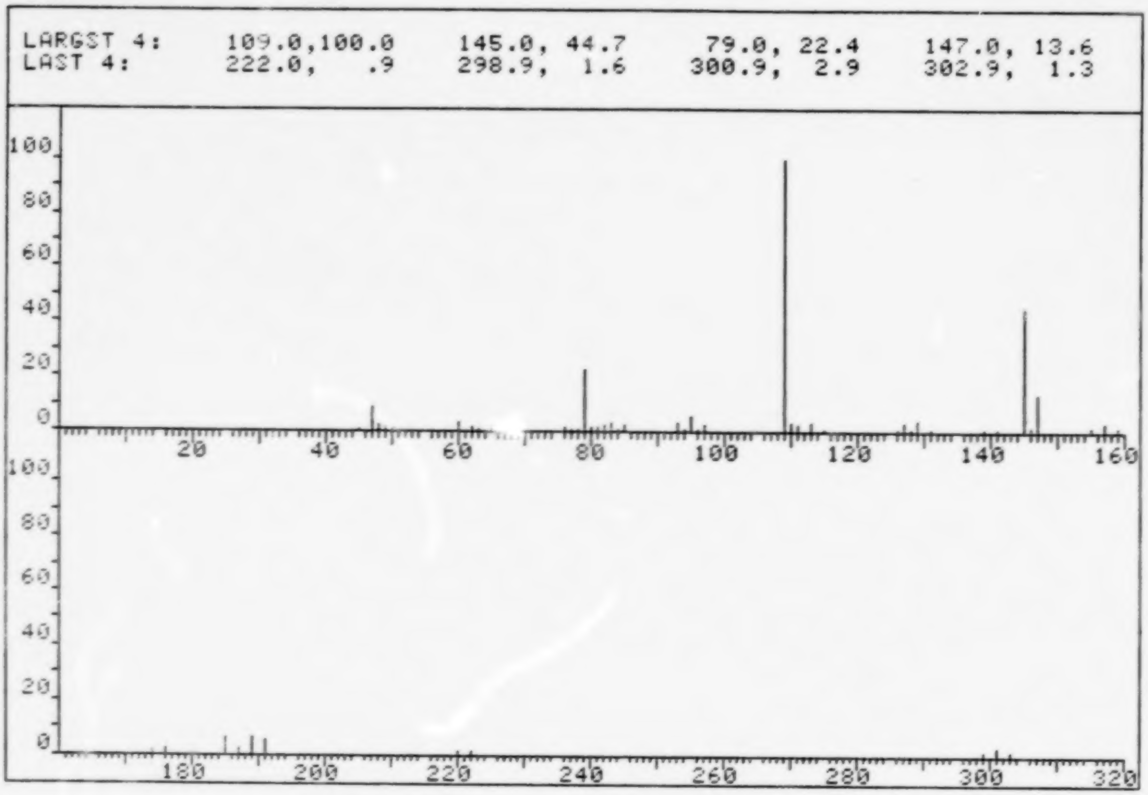


Figure D-68. Calibration curve for the determination of phosphoric triamide, hexamethyl- by GC/MS.

Range: 25 - 250 ng
Correlation coefficient: 0.991
Slope: 1.6×10^{-2}
y-Intercept: 1.5×10^{-1}



```
LARGST 4:    109.0,100.0    145.0, 44.7     79.0, 22.4    147.0, 13.6
LAST 4:      222.0,    .9   298.9,  1.6    300.9,  2.9    302.9,  1.3
```

Figure D-69. Mass spectrum for phosphoric acid, 1,2-dibromo-2,2-dichloroethyl dimethyl ester.

TABLE D-30. KEY IONS AND RELATIVE ABUNDANCES FOR PHOSPHORIC ACID, 1,2-DIBROMO-2,2-DICHLOROETHYL DIMETHYL ESTER

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
45	.9	81	1.7	115	.8	174	1.1
47	9.3	82	2.6	126	.9	176	1.8
48	2.3	83	3.8	127	3.4	185	6.4
49	1.8	84	1.2	128	1.2	187	2.3
50	.9	85	2.4	129	4.2	189	5.9
60	3.1	93	3.1	131	1.1	191	5.5
62	1.4	94	.9	139	.9	220	1.3
63	1.3	95	5.8	141	1.2	222	.9
65	1.7	96	1.1	145	44.7	299	1.6
76	1.4	97	2.0	146	1.3	301	2.9
77	1.0	109	100.0	147	13.6	303	1.3
78	1.2	110	3.3	155	1.9		
79	22.4	111	2.7	157	3.4		
80	1.8	113	3.3	159	1.6		

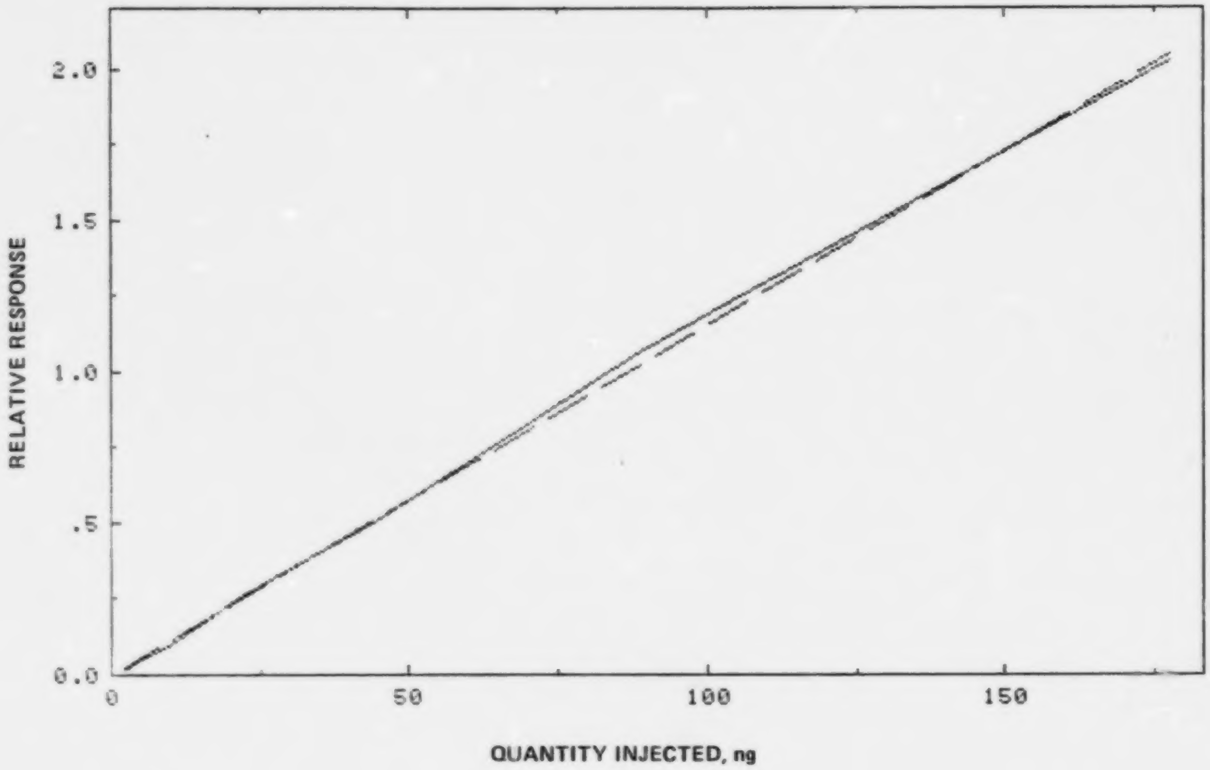
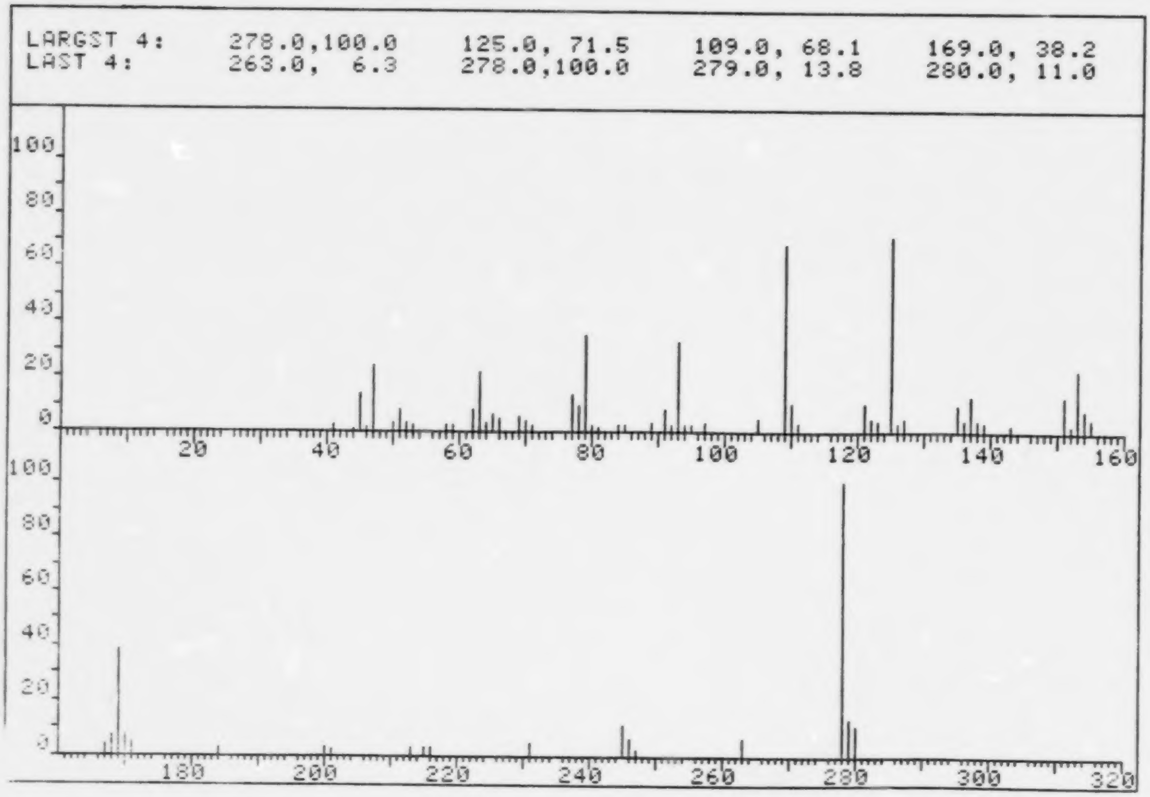


Figure D-70. Calibration curve for the determination of phosphoric acid, 1,2-dibromo-2,2-dichloroethyl dimethyl ester by GC/MS.

Range: 4.4 - 180 ng
Correlation coefficient: 0.999
Slope: 1.2×10^{-2}
y-Intercept: -1.1×10^{-3}



```
LARGST 4:     278.0,100.0     125.0, 71.5     109.0, 68.1     169.0, 38.2
LAST 4:       263.0,   6.3    278.0,100.0     279.0, 13.8     280.0, 11.0
```

Figure D-71. Mass spectrum for phosphorothioic acid, O,O-dimethyl-, O-(4-(methylthio)-m-tolyl) ester.

TABLE D-31. KEY IONS AND RELATIVE ABUNDANCES FOR PHOSPHOROTHIOIC ACID, *O*,*O*-DIMETHYL-, *O*-(4-(METHYLTHIO)-*m*-TOLYL) ESTER

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	2.1	77	13.8	121	10.8	168	7.1
45	13.8	78	9.9	122	5.0	169	38.2
46	1.9	79	35.4	123	4.1	170	8.0
47	24.1	80	2.1	125	71.5	171	4.3
50	3.0	81	1.9	126	3.0	184	2.4
51	7.8	84	2.4	127	5.2	200	2.6
52	3.4	85	2.1	135	10.1	201	2.1
53	2.1	89	3.0	136	4.1	213	2.6
58	2.6	91	8.4	137	12.5	215	2.6
59	2.4	92	2.2	138	4.3	216	2.4
62	8.4	93	33.2	139	3.5	231	4.8
63	22.0	94	2.2	143	2.4	245	10.8
64	3.5	95	2.6	151	12.9	246	6.0
65	6.5	97	3.5	152	2.6	247	2.2
66	5.0	105	4.8	153	22.6	263	6.3
69	6.0	109	68.1	154	8.2	278	100.0
70	4.1	110	10.4	155	4.7	279	13.8
71	2.6	111	3.5	167	3.9	280	11.0

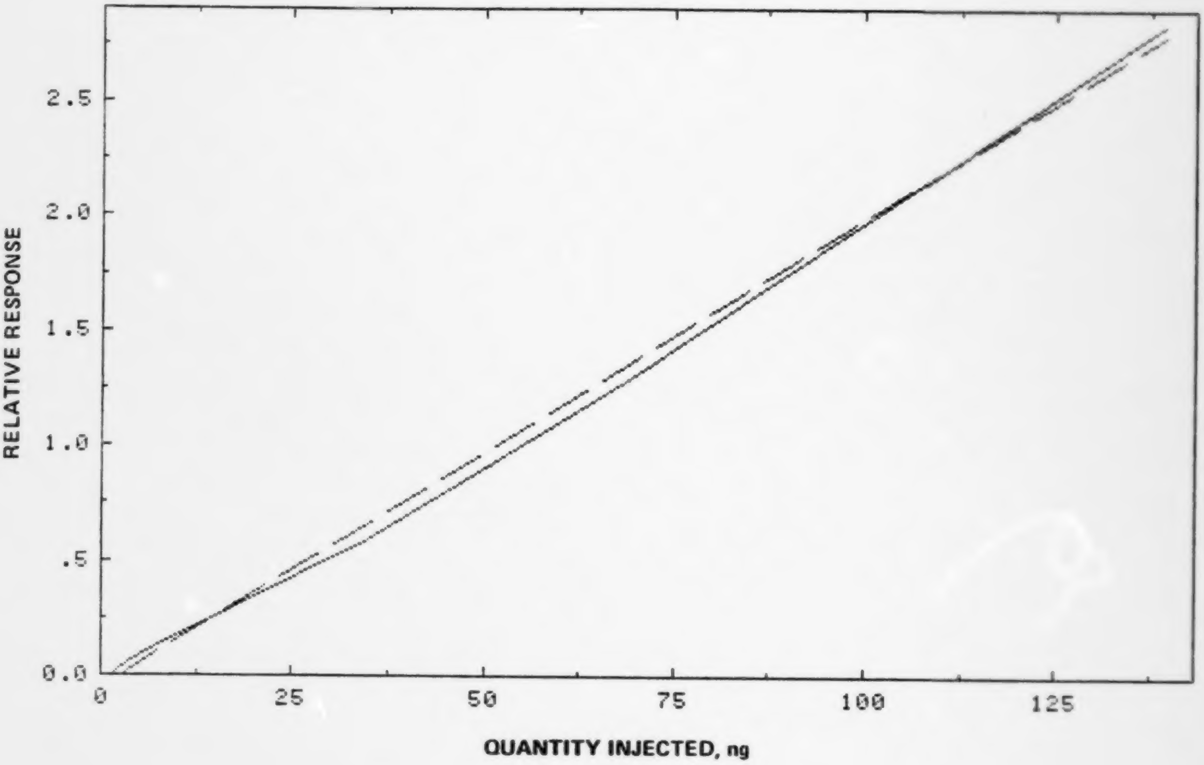
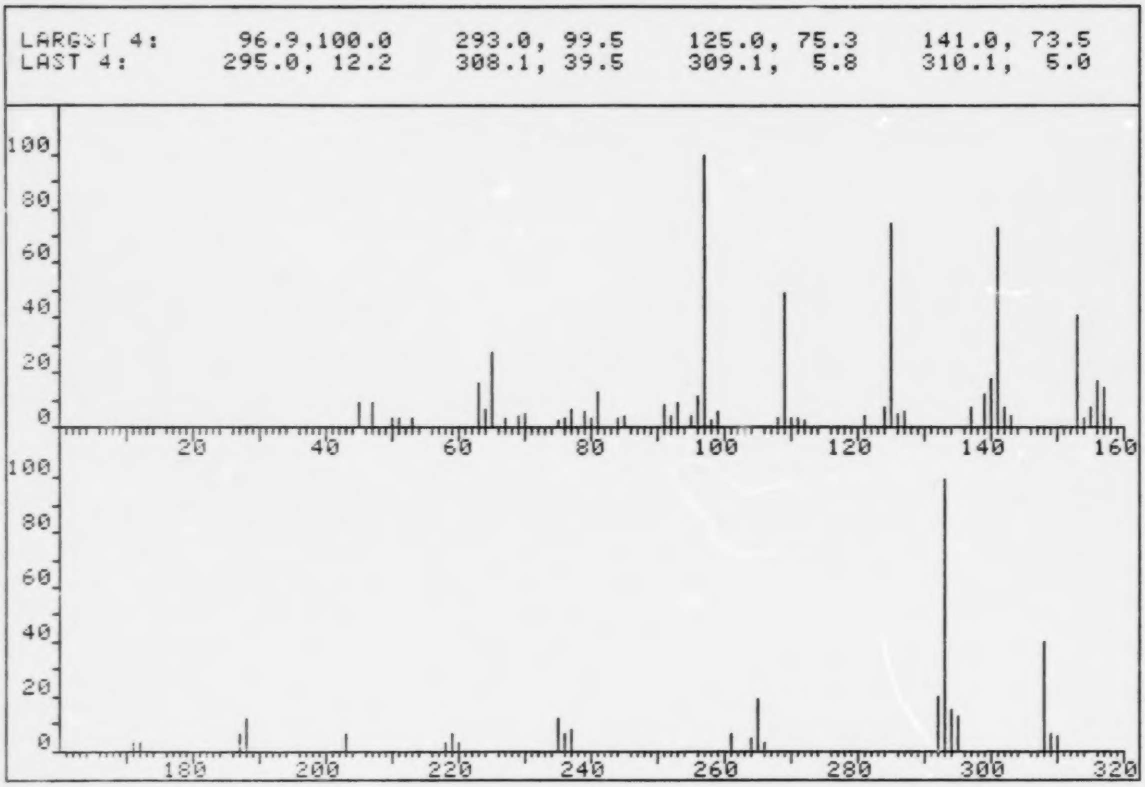


Figure D-72. Calibration curve for the determination of phosphorothioic acid, _O_,_O_-dimethyl-, _O_-(4-(methylthio)-_m_-tolyl) ester by GC/MS.

Range: 7 - 140 ng
Correlation coefficient: 0.999
Slope: 2.0×10^{-2}
y-Intercept: -4.8×10^{-2}

TABLE D-32. KEY IONS AND RELATIVE ABUNDANCES FOR PHOSPHOROTHIOIC ACID, *O*,*O*-DIETHYL, *O*-(*p*-(METHYLSULFINYL) PHENYL) ESTER

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
45	9.3	85	4.0	127	5.8	218	2.7
47	9.0	91	7.7	137	7.4	219	5.8
50	2.9	92	4.0	139	12.2	220	2.9
51	3.2	93	8.8	140	18.3	235	11.4
53	2.9	95	4.0	141	73.5	236	6.4
63	16.2	96	11.4	142	7.2	237	8.2
64	6.4	97	100.0	143	4.2	261	5.8
65	27.6	98	2.7	153	41.1	264	4.8
67	2.9	99	5.6	154	2.9	265	18.8
69	4.5	108	2.9	155	7.2	266	2.7
70	5.0	109	49.3	156	17.2	292	19.6
75	2.7	110	3.4	157	14.9	293	99.5
76	3.7	111	3.2	158	3.7	294	14.9
77	6.6	112	2.7	171	2.9	295	12.2
79	5.8	121	4.2	172	3.4	308	39.5
80	3.2	124	7.4	187	6.1	309	5.8
81	13.0	125	75.3	188	11.7	310	5.0
84	2.9	126	4.8	203	5.8		



```
LARGST 4:     96.9,100.0     293.0, 99.5     125.0, 75.3     141.0, 73.5
LAST 4:      295.0, 12.2     308.1, 39.5     309.1,  5.8     310.1,  5.0
```

Figure D-73. Mass spectrum for phosphorothioic acid, O,O-diethyl O-(p-(methylsulfinyl) phenyl) ester.

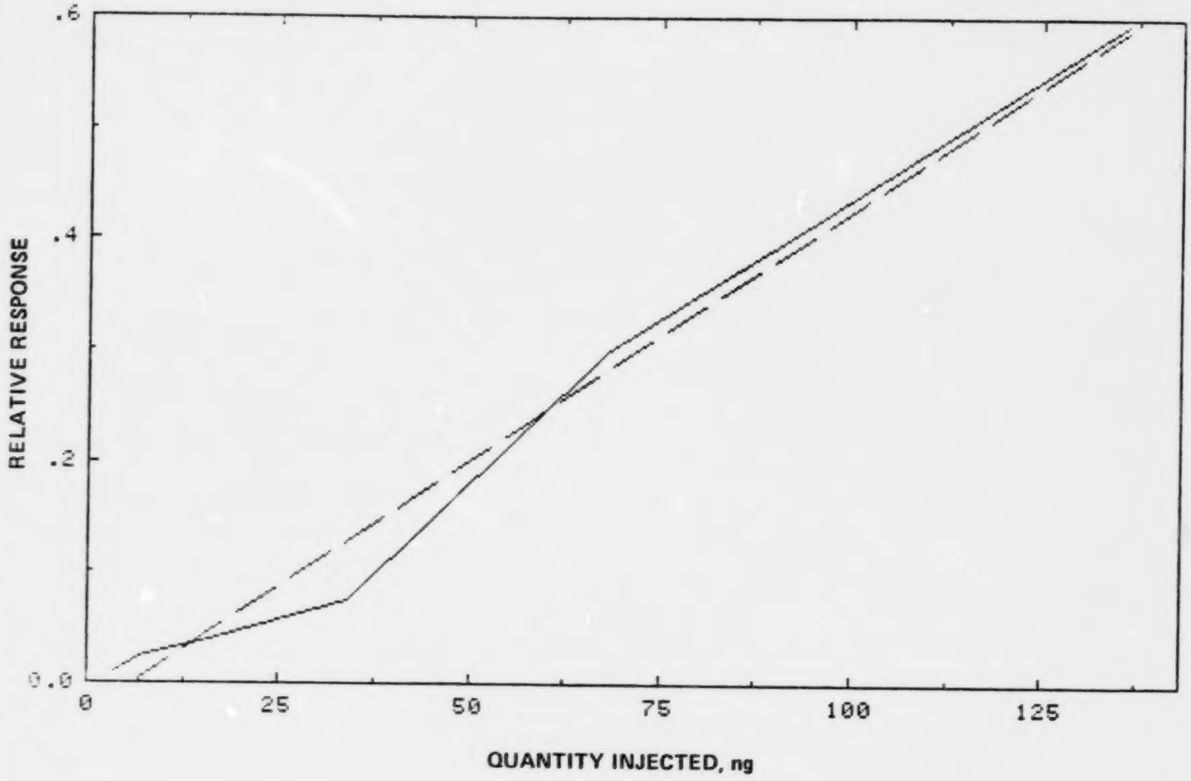


Figure D-74. Calibration curve for the determination of phosphorothioic acid, O,O-diethyl O-(p-(methylsulfinyl) phenyl) ester by GC/MS.

Range: 17 - 140 ng
Correlation coefficient: 0.993
Slope: 4.5×10^{-3}
y-Intercept: -2.6×10^{-2}

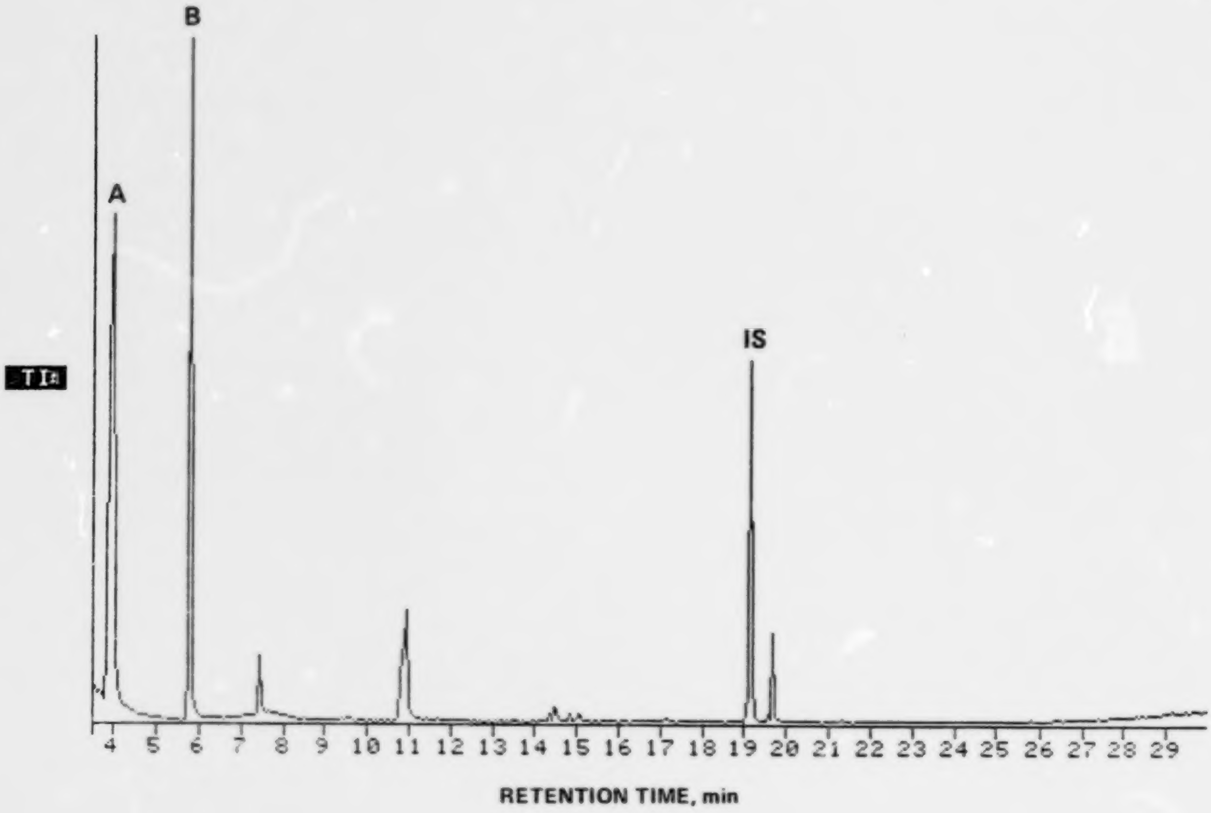


Figure D-75. Chromatogram by GC/MS for

(A) propionitrile, 2-hydroxy- (t_R = 4.0 min) and
(B) hydroxylamine, N-nitroso-N-phenyl-, ammonium salt(t_R = 5.8 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

1 to 10 split ratio

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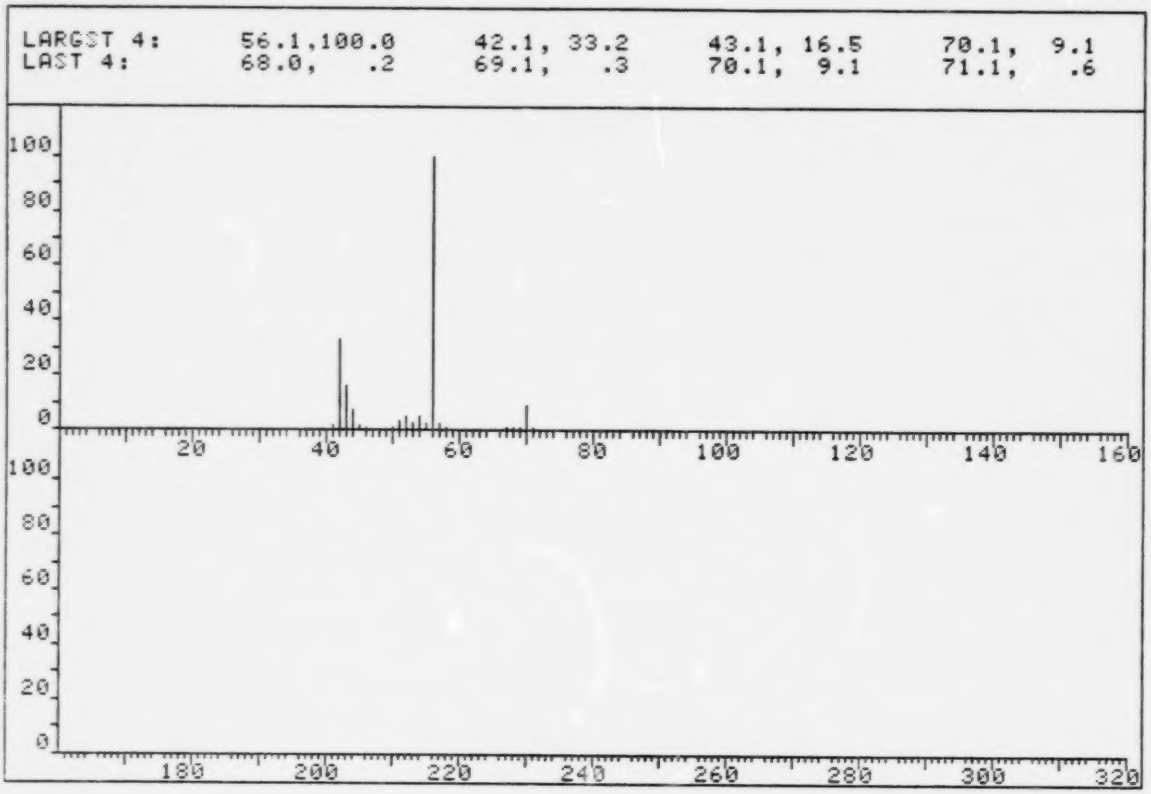


```
LARGST 4:      56.1,100.0      42.1, 33.2      43.1, 16.5      70.1,   9.1
LAST 4:        68.0,    .2     69.1,    .3     70.1,   9.1     71.1,    .6
```

Figure D-76. Mass spectrum for propionitrile, 2-hydroxy-.

TABLE D-33. KEY IONS AND RELATIVE ABUNDANCES FOR PROPIONITRILE, 2-HYDROXY-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	1.8	45	1.7	53	2.1	57	2.7
42	33.2	50	.6	54	4.9	70	9.1
43	16.5	51	3.8	55	2.4	71	.6
44	7.2	52	5.0	56	100.0		

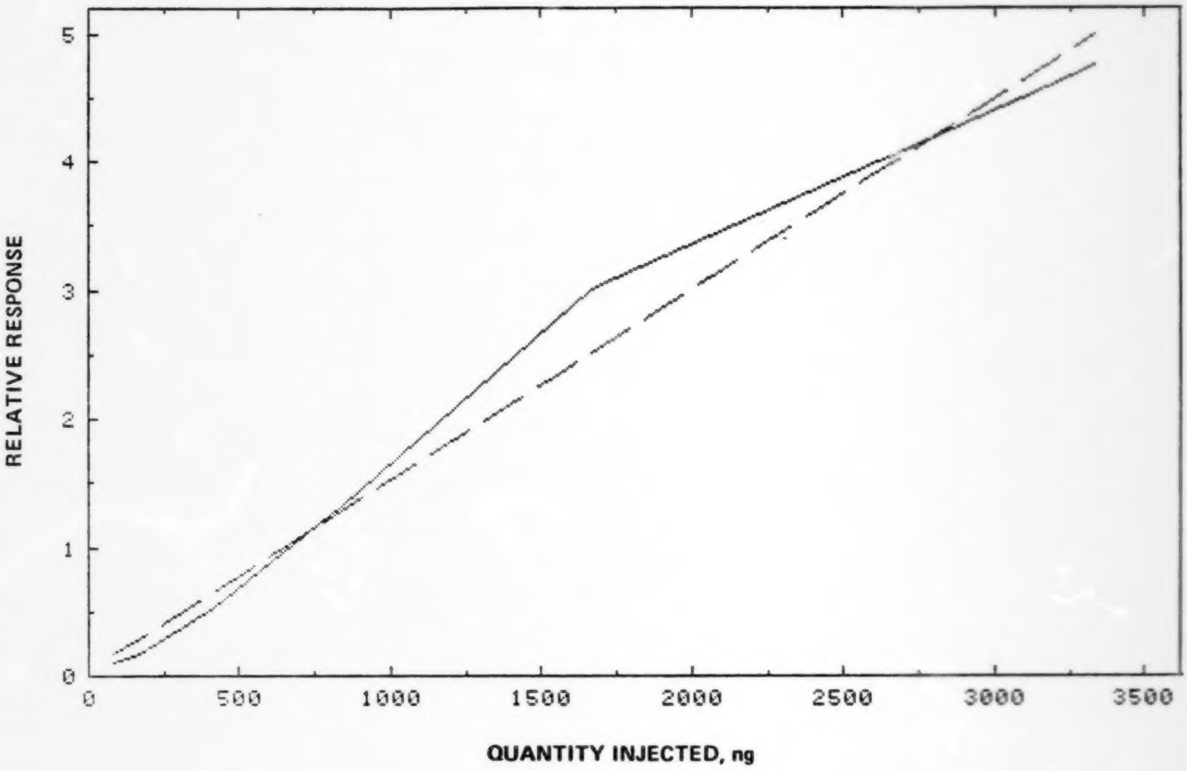
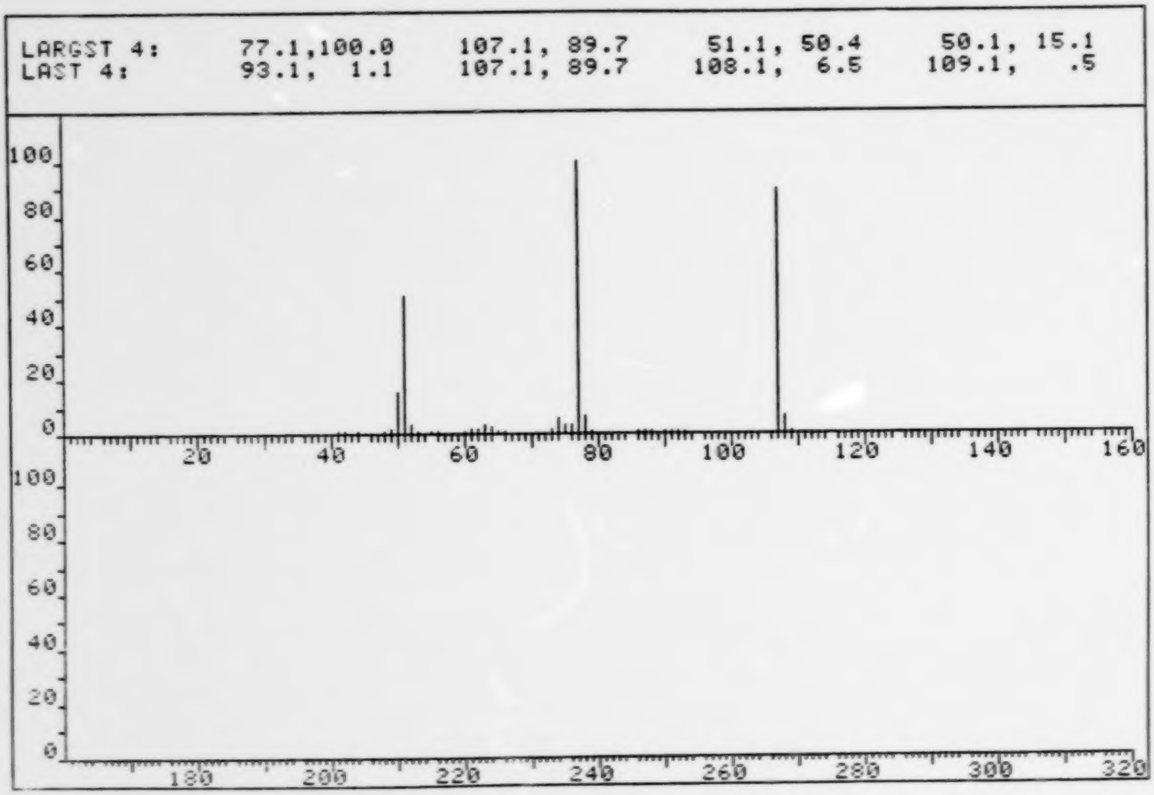


Figure D-77. Calibration curve for the determination of propionitrile, 2-hydroxy- by GC/MS.

Range: 83 - 3300 ng
Correlation coefficient: 0.990
Slope: 1.5×10^{-3}
y-Intercept: 3.9×10^{-2}



```
LARGST 4:     77.1,100.0     107.1, 89.7      51.1, 50.4      50.1, 15.1
LAST 4:       93.1,   1.1    107.1, 89.7     108.1,  6.5     109.1,   .5
```

Figure D-78. Mass spectrum for hydroxylamine, N-nitroso-N-phenyl-, ammonium salt.

TABLE D-34. KEY IONS AND RELATIVE ABUNDANCES FOR HYDROXYLAMINE, N-NITROSO-N-PHENYL-, AMMONIUM SALT

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
49	1.6	62	1.4	74	5.9	91	.5
50	15.1	63	2.9	75	3.0	93	1.1
51	50.4	64	2.2	76	3.0	107	89.7
52	3.1	65	.7	77	100.0	108	6.5
61	1.3	73	1.5	78	6.5		

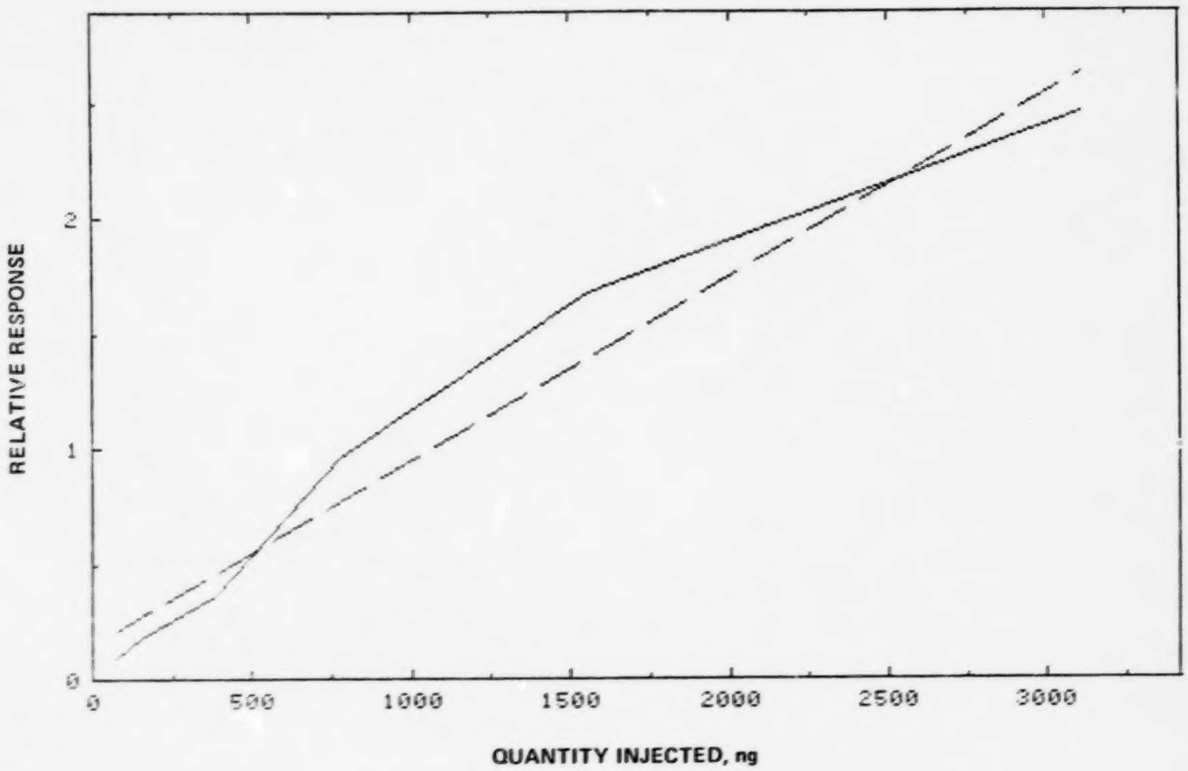


Figure D-79. Calibration curve for the determination of hydroxylamine, N-nitroso-N-phenyl-, ammonium salt by GC/MS.

Range: 160 - 3100 ng
Correlation coefficient: 0.979
Slope: 8.0×10^{-4}
y-Intercept: 1.5×10^{-1}

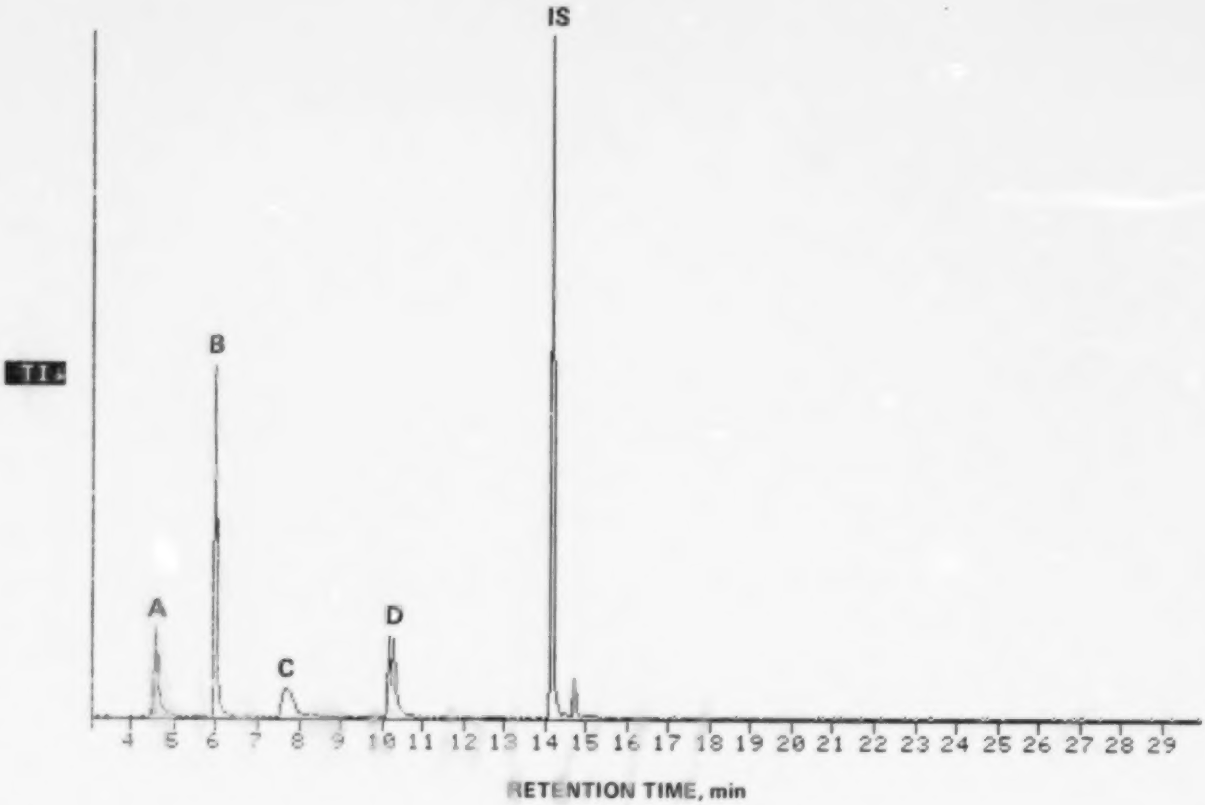
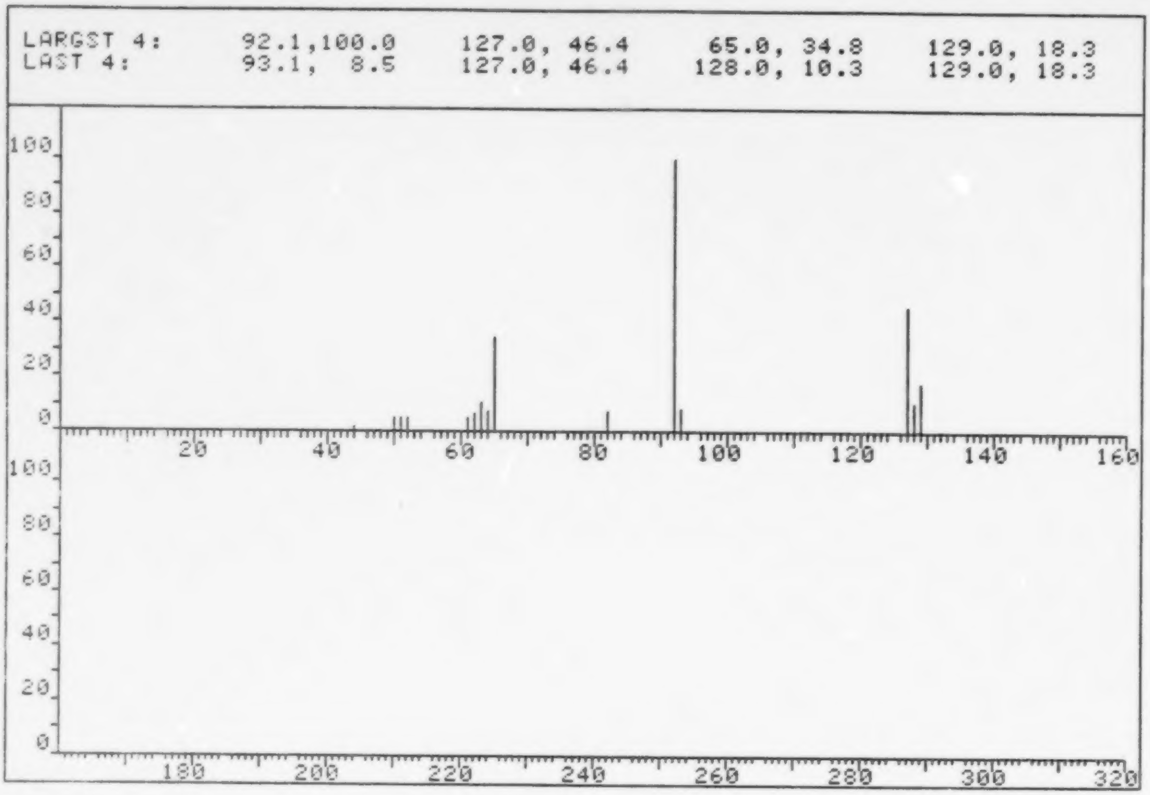


Figure D-80. Chromatogram by GC/MS for

(A) pyridine, 3-chloromethyl-, hydrochloride(t_R = 4.7 min),
(B) o-anisidine hydrochloride(t_R = 6.0 min),
(C) hydroquinone(t_R = 7.7 min), and
(D) o-phenylenediamine, 4-chloro- (t_R = 10.3 min).

Temperature program:
80 °C, 3 min isothermal
80 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

1 to 10 split ratio



```
LARGST 4:     92.1,100.0    127.0, 46.4     65.0, 34.8    129.0, 18.3
LAST 4:       93.1,   8.5   127.0, 46.4    128.0, 10.3    129.0, 18.3
```

Figure D-81. Mass spectrum for pyridine, 3-chloromethyl-, hydrochloride.

TABLE D-35. KEY IONS AND RELATIVE ABUNDANCES FOR PYRIDINE, 3-CHLOROMETHYL-, HYDROCHLORIDE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	1.8	61	4.9	65	34.8	127	46.4
50	4.9	62	6.7	82	7.6	128	10.3
51	5.4	63	10.3	92	100.0	129	18.3
52	5.4	64	7.6	93	8.5		

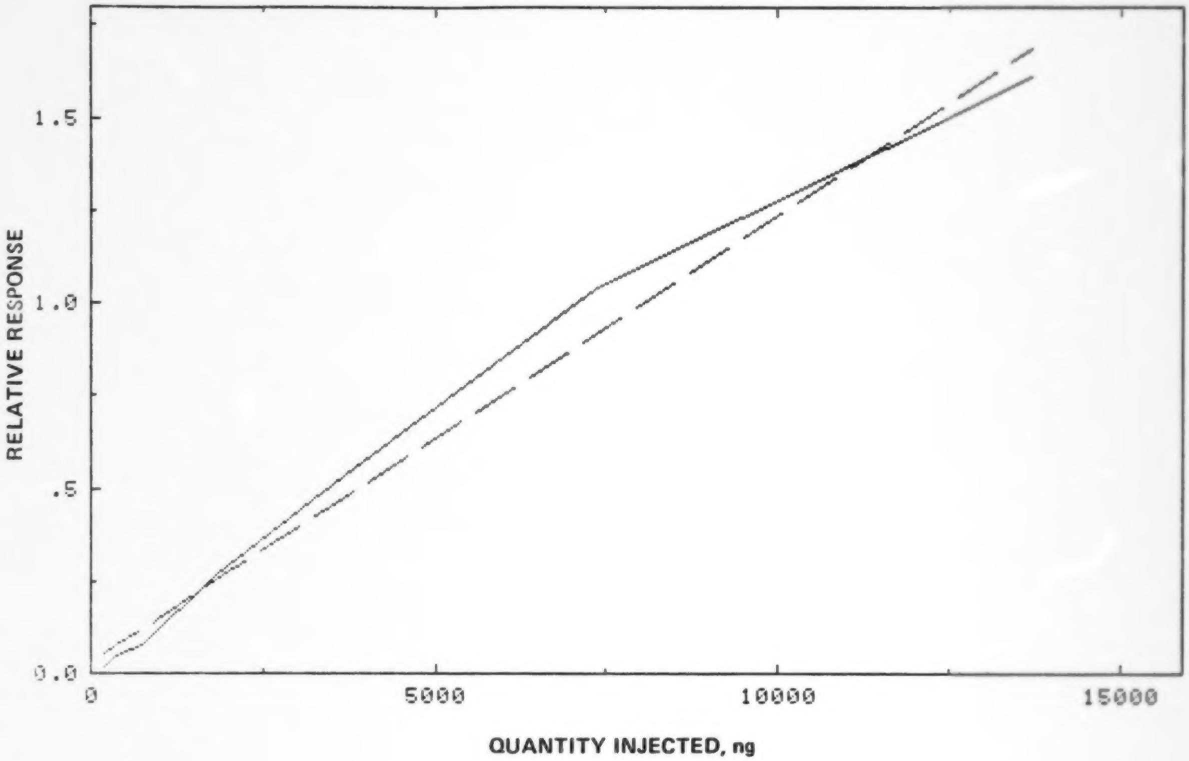
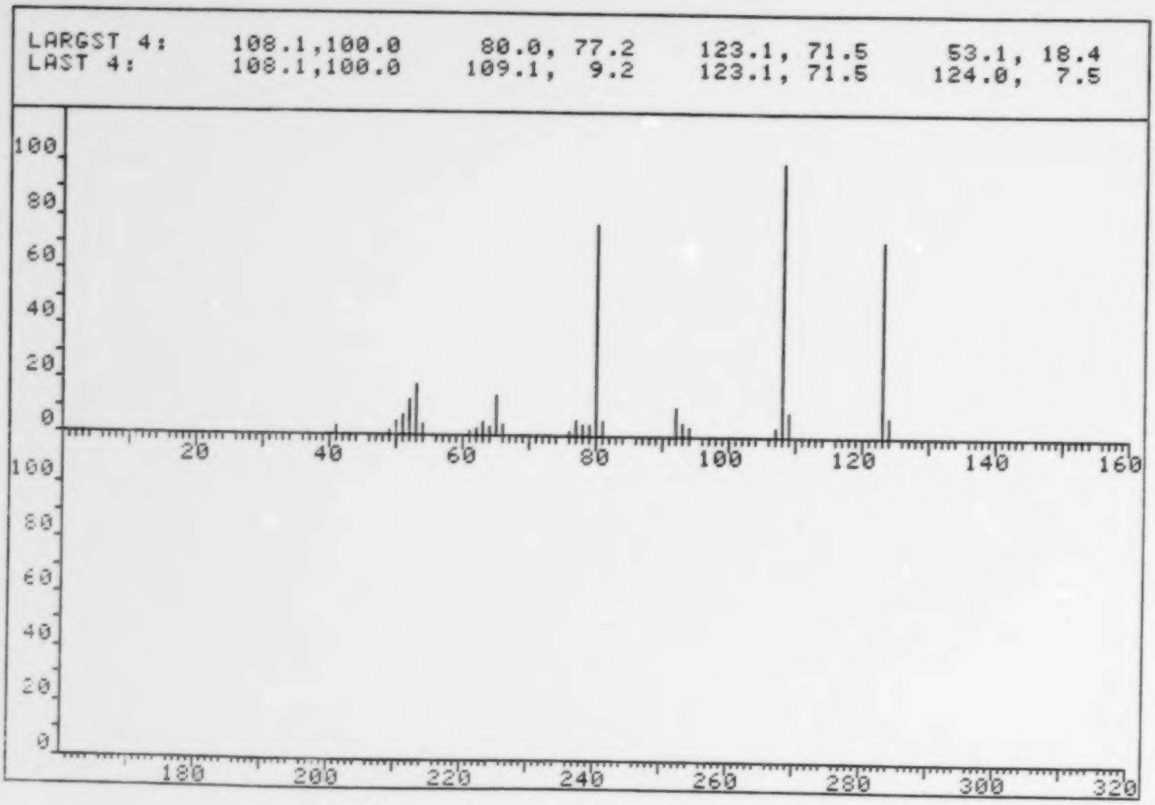


Figure D-82. Calibration curve for the determination of pyridine, 3-chloromethyl-, hydrochloride by GC/MS.

Range: 740 - 14000 ng
Correlation coefficient: 0.989
Slope: 1.1×10^{-4}
y-Intercept: 4.7×10^{-2}



```
LARGST 4:     108.1,100.0      80.0, 77.2     123.1, 71.5      53.1, 18.4
LAST 4:       108.1,100.0     109.1,  9.2     123.1, 71.5     124.0,  7.5
```

Figure D-83. Mass spectrum for o-anisidine hydrochloride.

TABLE D-36. KEY IONS AND RELATIVE ABUNDANCES FOR *o*-ANISIDINE HYDROCHLORIDE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	3.2	61	2.0	77	5.7	94	3.2
49	1.7	62	2.7	78	4.4	107	3.2
50	5.2	63	5.2	79	4.2	108	100.0
51	7.4	64	3.0	80	77.2	109	9.2
52	12.7	65	14.4	81	5.7	123	71.5
53	18.4	66	4.2	92	10.4	124	7.5
54	4.4	76	1.8	93	5.4		

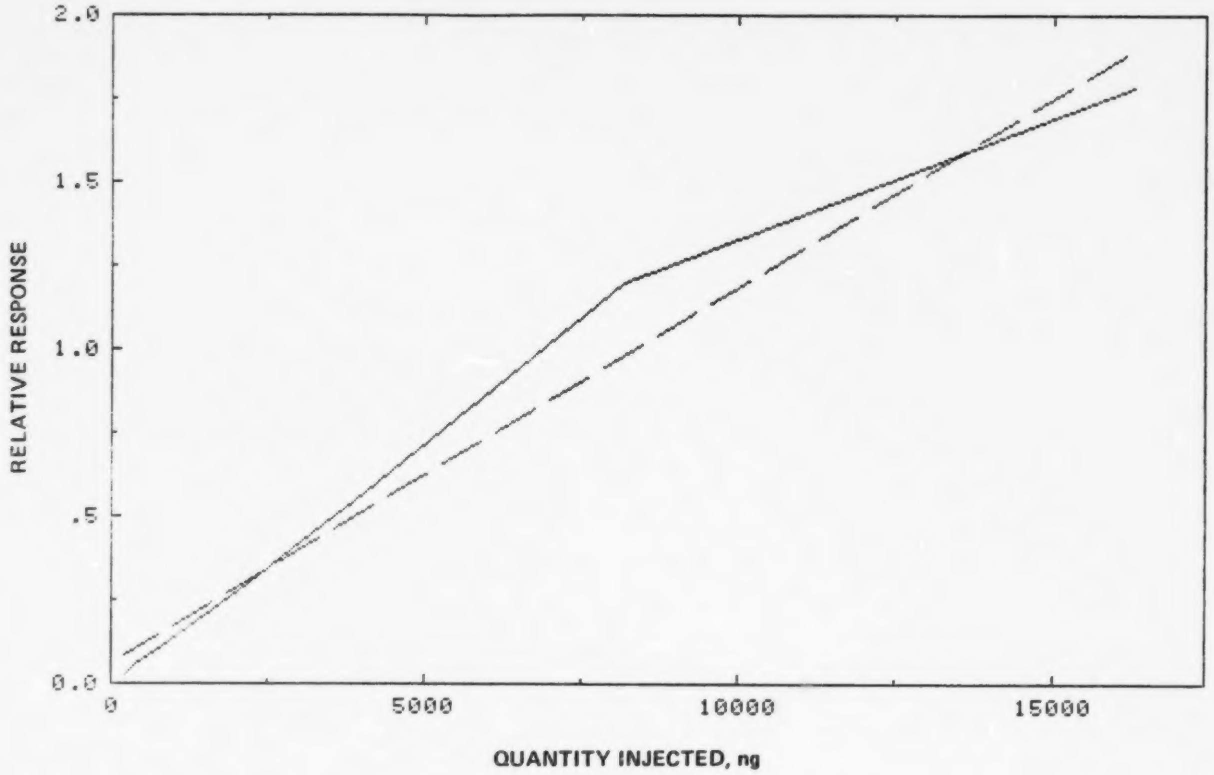
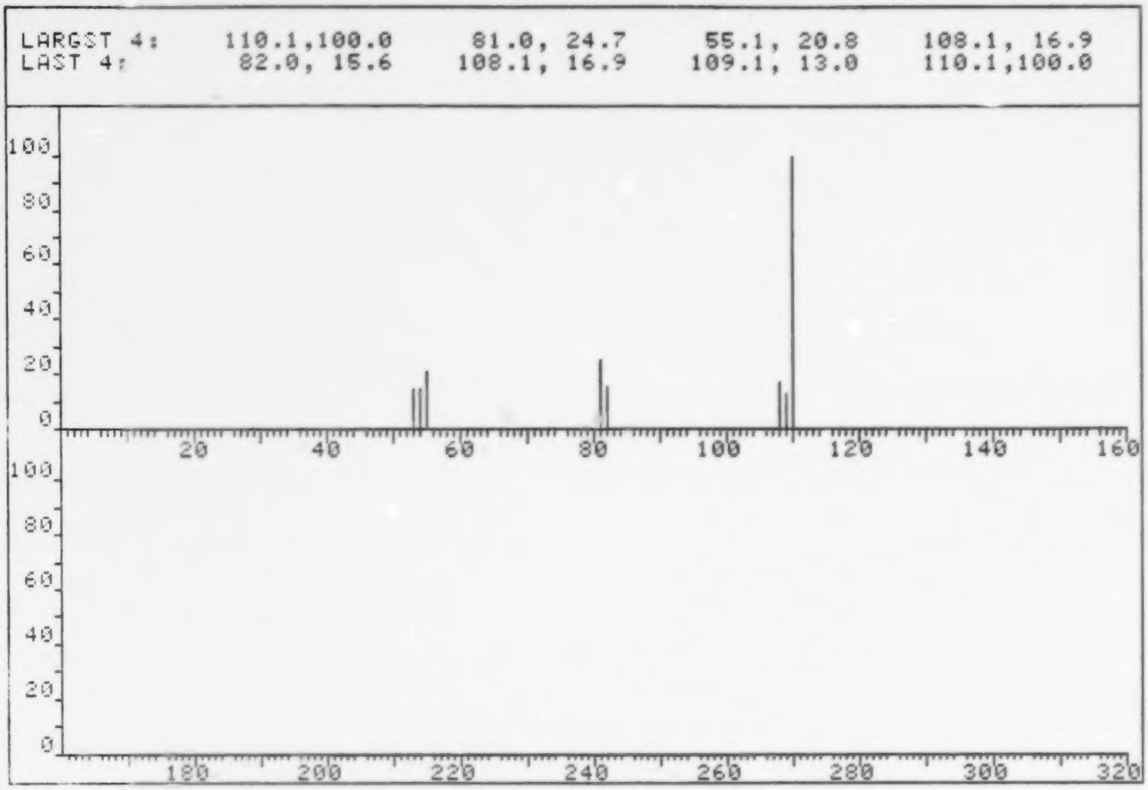


Figure D-84. Calibration curve for the determination of o-anisidine hydrochloride by GC/MS.

Range: 820 - 16000 ng
Correlation coefficient: 0.987
Slope: 1.1×10^{-4}
y-Intercept: 6.0×10^{-2}



```
LARGST 4:     110.1,100.0      81.0, 24.7      55.1, 20.8     108.1, 16.9
LAST 4:        82.0, 15.6     108.1, 16.9     109.1, 13.0     110.1,100.0
```

Figure D-85. Mass spectrum for hydroquinone.

TABLE D-37. KEY IONS AND RELATIVE ABUNDANCES FOR HYDROQUINONE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
53	14.3	55	20.8	82	15.6	109	13.0
54	14.3	81	24.7	108	16.9	110	100.0

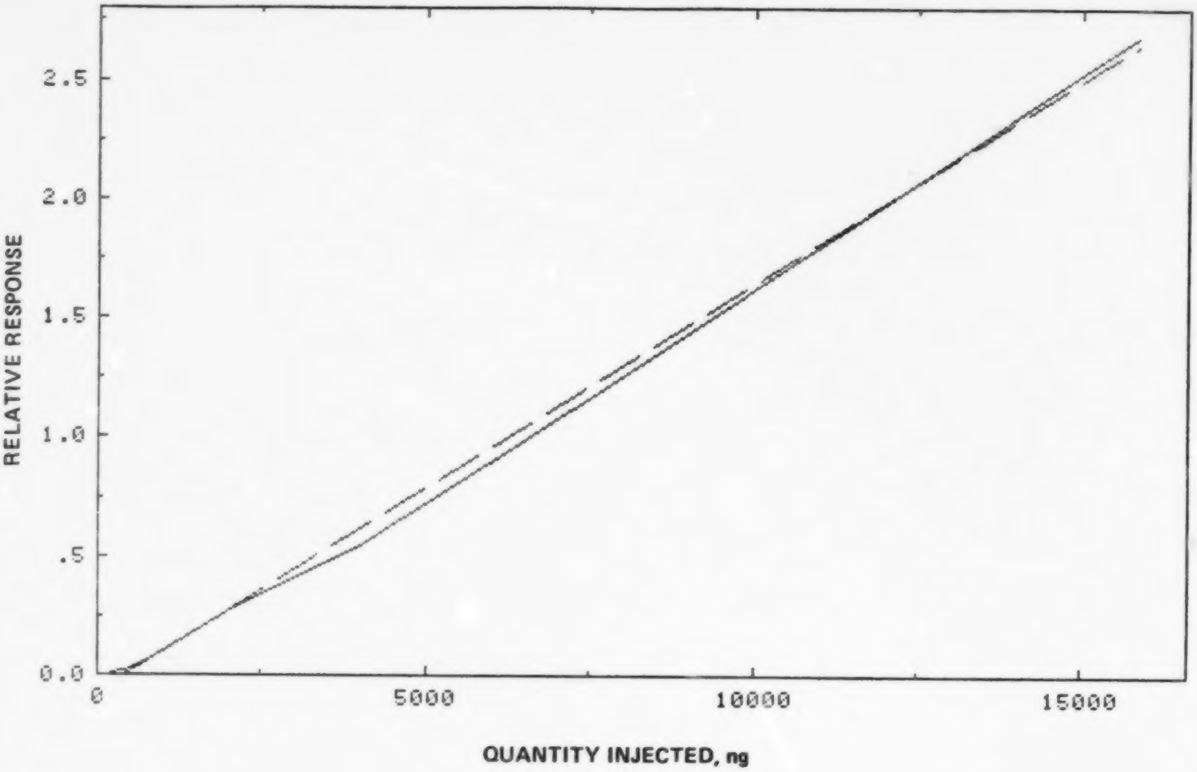


Figure D-86. Calibration curve for the determination of hydroquinone by GC/MS.

Range: 790 - 16000 ng
Correlation coefficient: 0.999
Slope: 1.7×10^{-4}
y-Intercept: -7.1×10^{-2}

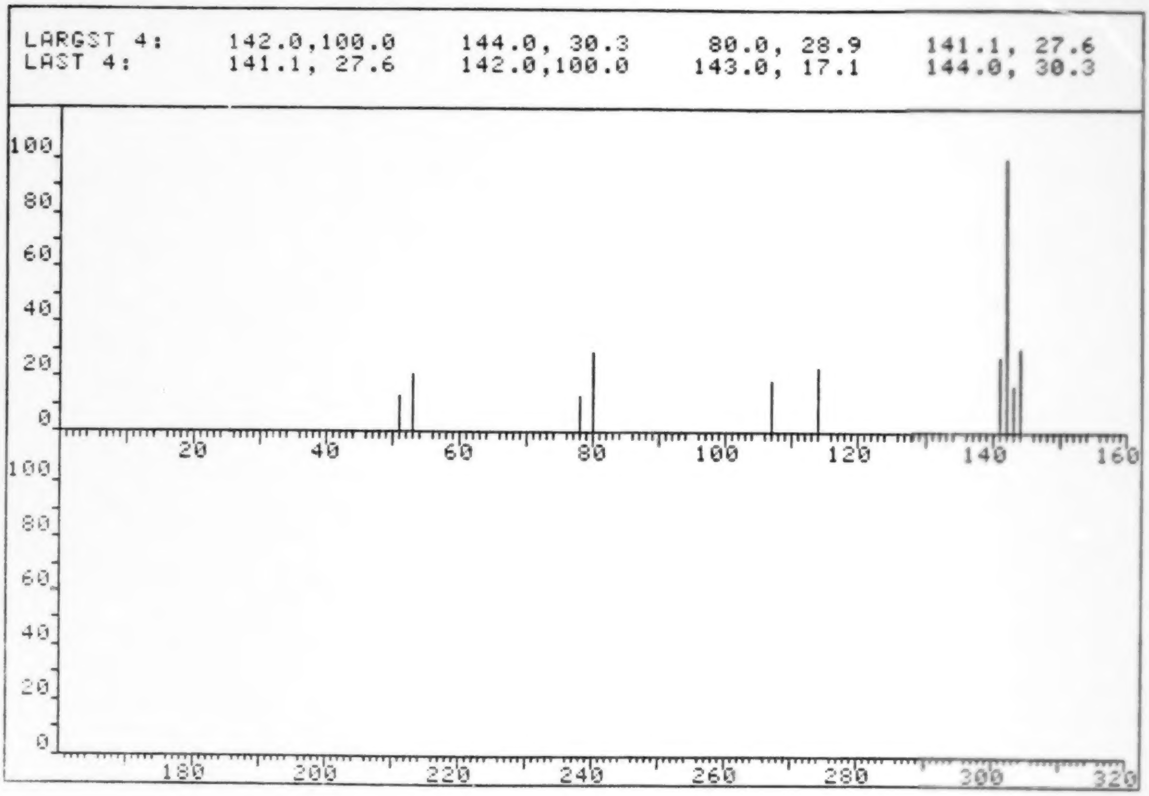


Figure D-87. Mass spectrum for o-phenylenediamine, 4-chloro- .

TABLE D-38. KEY IONS AND RELATIVE ABUNDANCES FOR o-PHENYLENEDIAMINE, 4-CHLORO-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
51	13.2	80	28.9	141	27.6	144	30.3
53	21.1	107	18.4	142	100.0		
78	13.2	114	23.7	143	17.1		

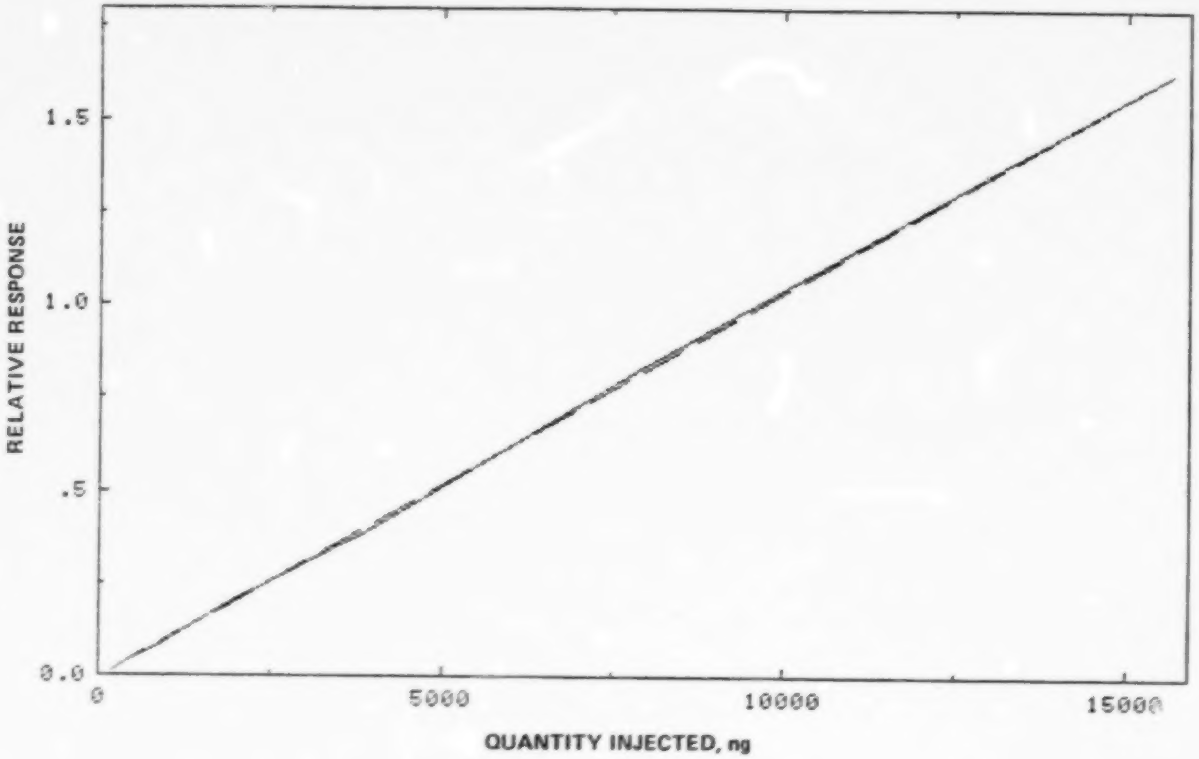


Figure D-88. Calibration curve for the determination of o-phenylenediamine, 4-chloro- by GC/MS.

Range: 200 - 16000 ng
Correlation coefficient: 0.999
Slope: 1.0×10^{-4}
y-Intercept: -7.6×10^{-3}

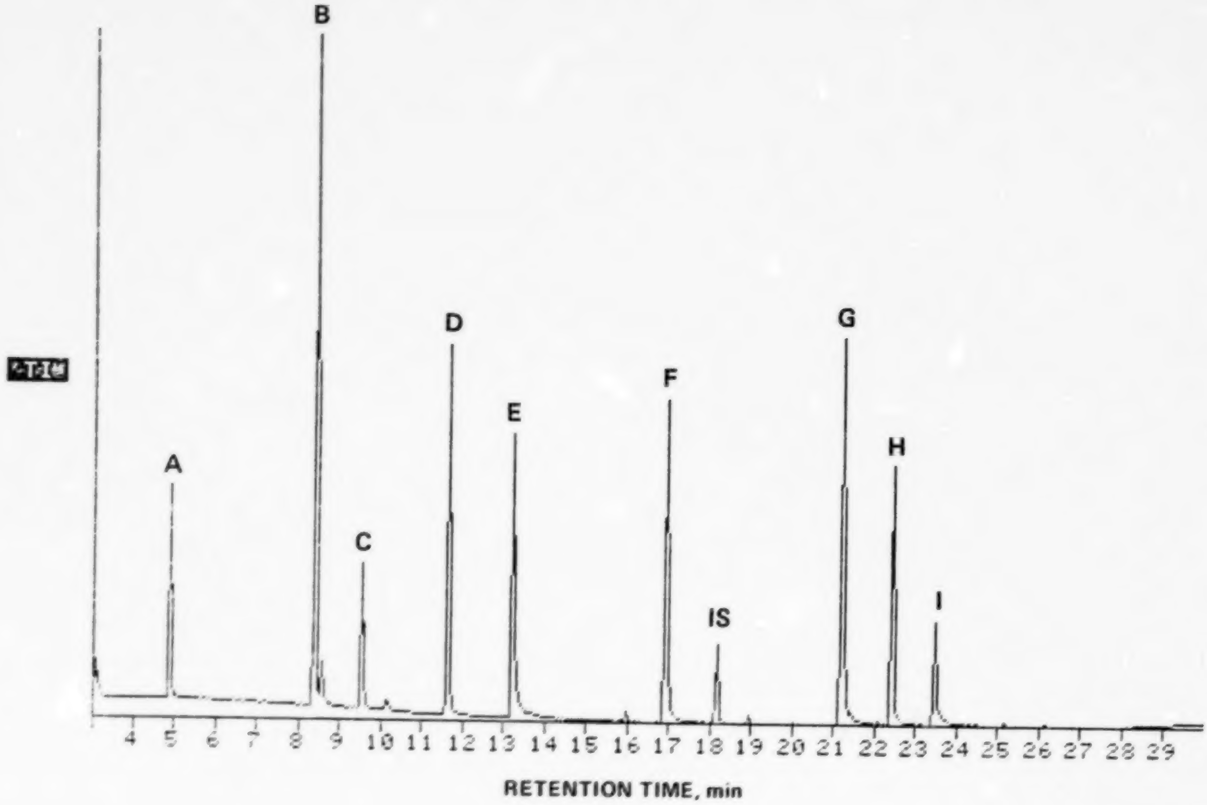


Figure D-89. Chromatogram by GC/MS for

- (A) styrene(t_R = 4.9 min),
- (B) o-toluidine(t_R = 8.36 min),
- (C) 2-oxetanone (2-oxepanone) (t_R = 9.5 min),
- (D) o-anisidine, 5-methyl- (t_R = 11.6 min),
- (E) toluene, 2,4-diamino- (t_R = 13.3 min),
- (F) benzonitrile, 3,5-dibromo-4-hydroxy- (t_R = 17.0 min),
- (G) aniline, p-(phenylazo)- (t_R = 21.2 min),
- (H) 3-amino-9-ethyl carbazole(t_R = 22.5 min), and
- (I) pyridine, 2,6-diamino-3-(phenylazo)-, monohydrochloride(t_R = 23.5 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

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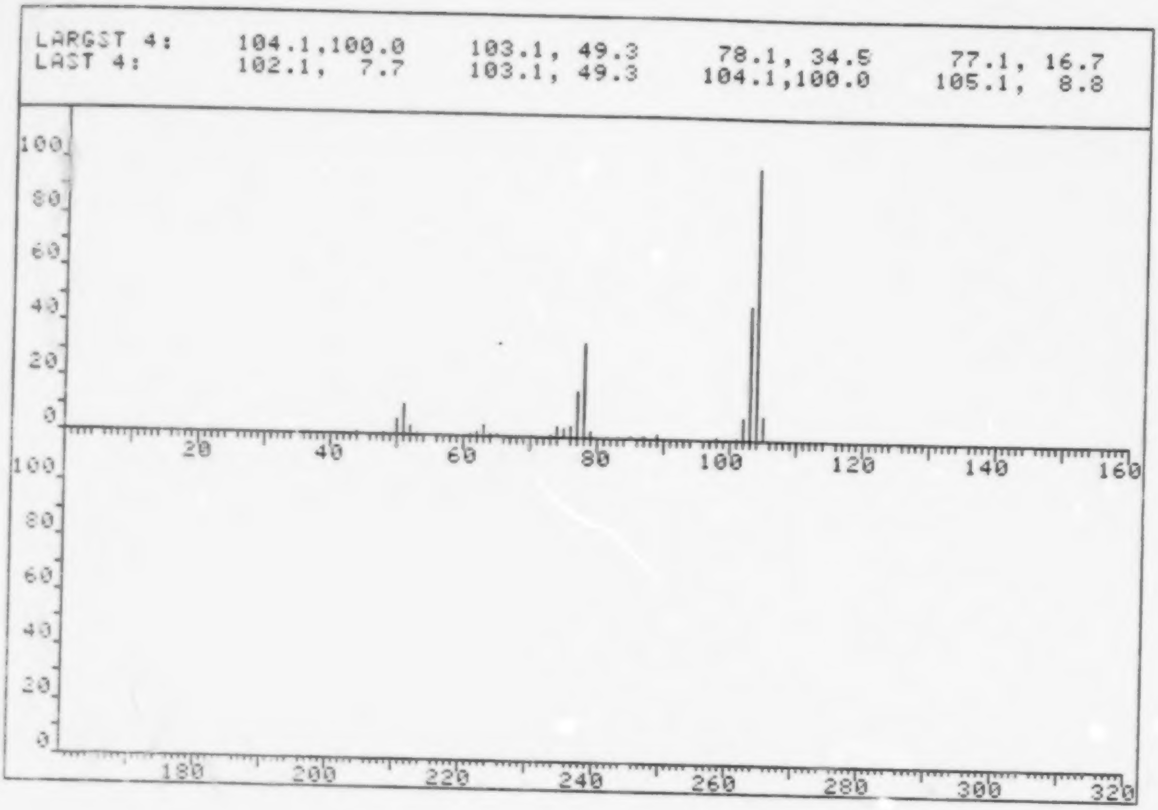


```
LARGST 4:     104.1,100.0     103.1, 49.3      78.1, 34.5      77.1, 16.7
LAST 4:       102.1,   7.7    103.1, 49.3     104.1,100.0     105.1,   8.8
```

Figure D-90. Mass spectrum for styrene.

TABLE D-39. KEY IONS AND RELATIVE ABUNDANCES FOR STYRENE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
48	.5	63	4.5	77	16.7	101	.8
50	6.0	64	.9	78	34.5	102	7.7
51	11.2	65	1.0	79	2.7	103	49.3
52	3.9	73	1.1	85	1.0	104	100.0
53	.9	74	3.9	87	.9	105	8.8
61	.9	75	3.0	89	1.8		
62	1.7	76	3.9	98	1.0		

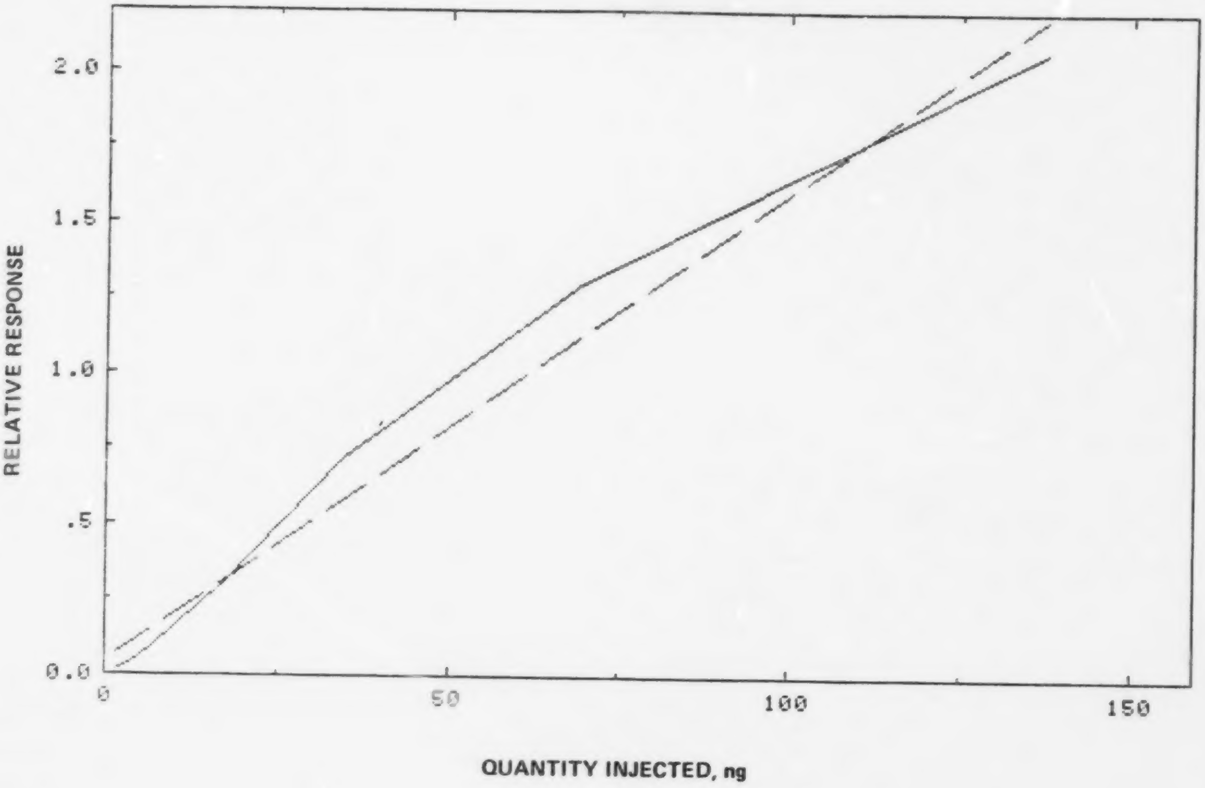
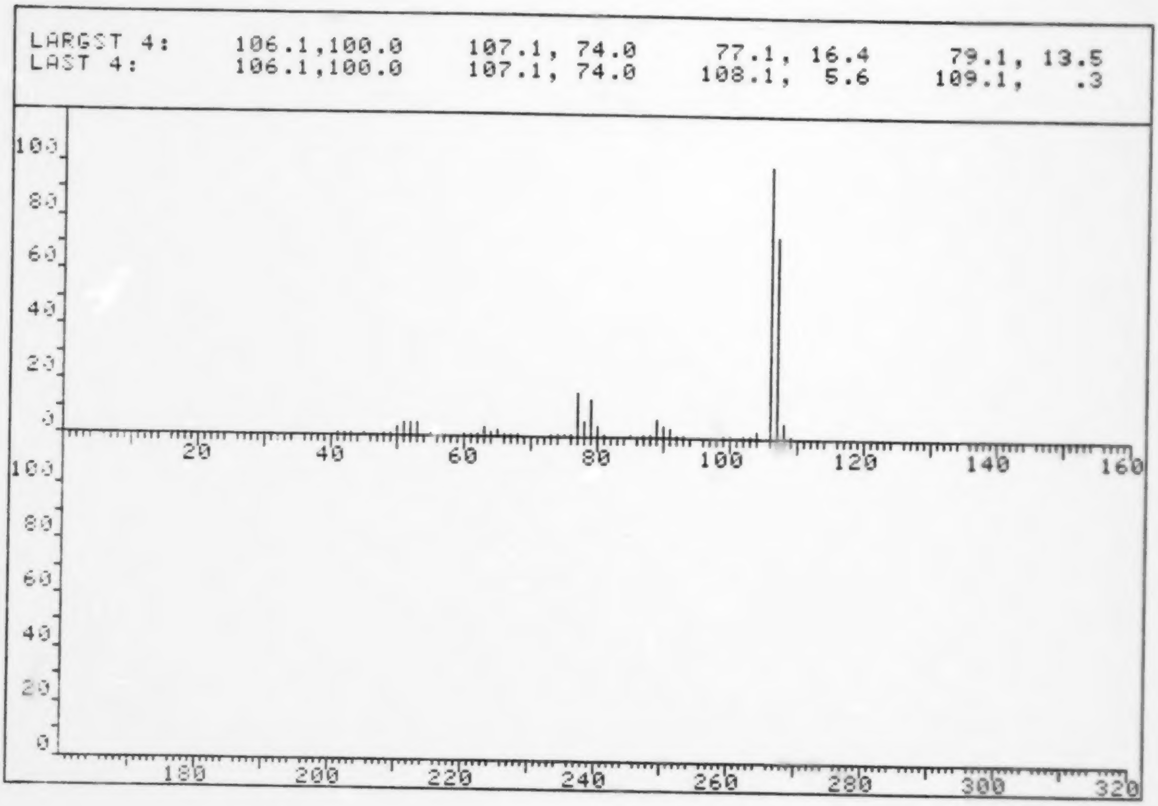


Figure D-91. Calibration curve for the determination of styrene by GC/MS.

Range: 12 - 140 ng
Correlation coefficient: 0.990
Slope: 1.6×10^{-2}
y-Intercept: 4.7×10^{-2}



```
LARGST 4:     106.1,100.0     107.1, 74.0      77.1, 16.4      79.1, 13.5
LAST 4:       106.1,100.0     107.1, 74.0     108.1,  5.6     109.1,   .3
```

Figure D-92. Mass spectrum for o-toluidine.

TABLE D-40. KEY IONS AND RELATIVE ABUNDANCES FOR o-TOLUIDINE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	1.2	63	3.1	77	16.4	92	.6
42	.9	64	1.3	78	5.5	104	2.6
50	3.1	65	2.4	79	13.5	106	100.0
51	4.9	66	1.2	80	4.4	107	74.0
52	5.2	67	.6	81	.7	108	5.6
53	5.1	74	1.0	89	6.8		
61	.5	75	.9	90	4.1		
62	1.2	76	1.2	91	3.0		

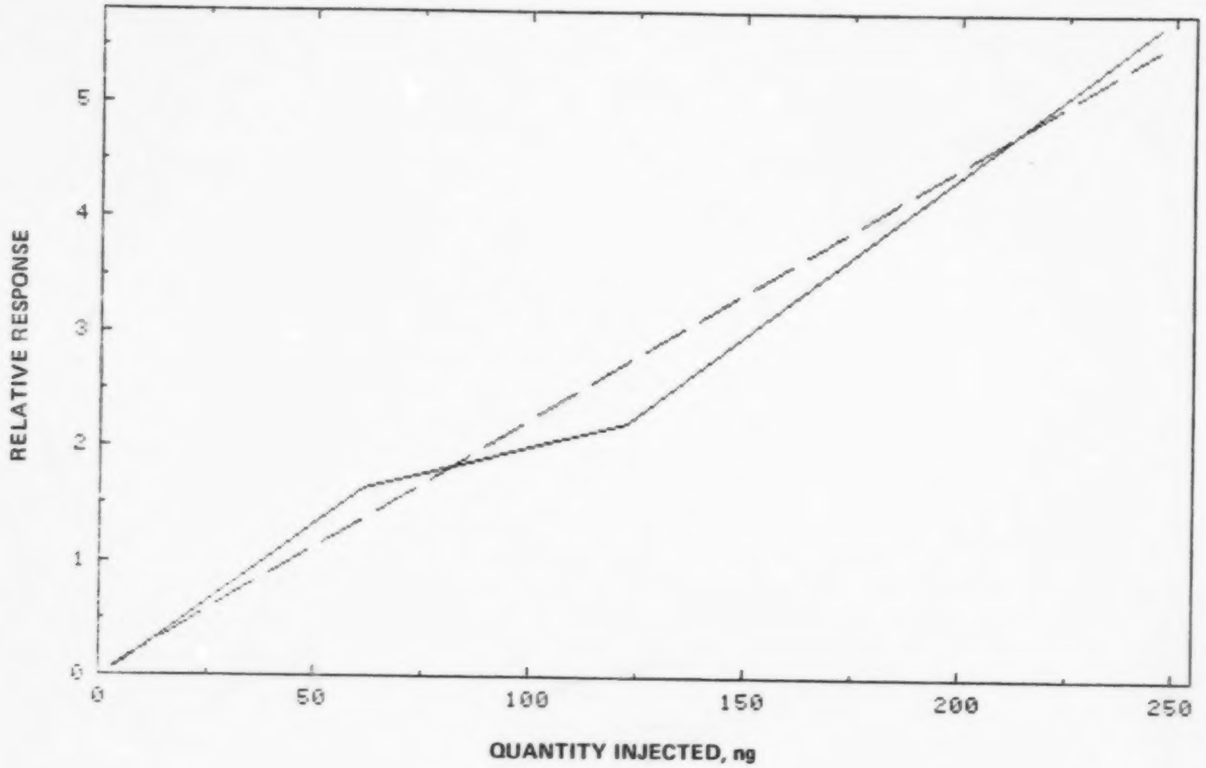


Figure D-93. Calibration curve for the determination of o-toluidine by GC/MS.

Range: 6.2 - 250 ng
Correlation coefficient: 0.991
Slope: 2.2×10^{-2}
y-Intercept: 1.3×10^{-2}

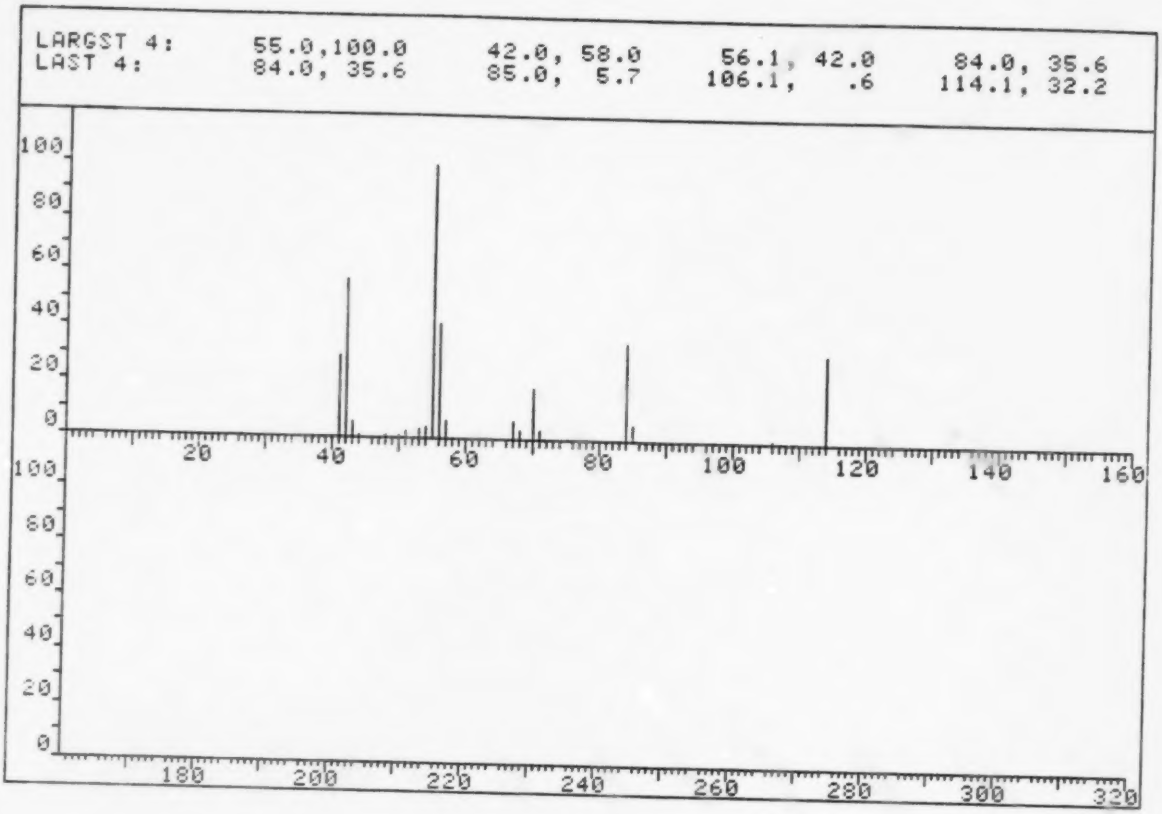

```
LARGST 4:     55.0,100.0      42.0, 58.0      56.1, 42.0      84.0, 35.6
LAST 4:       84.0, 35.6      85.0,  5.7     106.1,   .6     114.1, 32.2
```

Figure D-94. Mass spectrum for 2-oxepanone.

TABLE D-41. KEY IONS AND RELATIVE ABUNDANCES FOR 2-OXEPANONE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	30.0	50	.9	56	42.0	71	3.2
42	58.0	51	2.5	57	6.6	84	35.6
43	5.7	53	3.8	67	6.3	85	5.7
44	.9	54	4.4	68	3.5	106	.6
48	.9	55	100.0	70	18.6	114	32.2

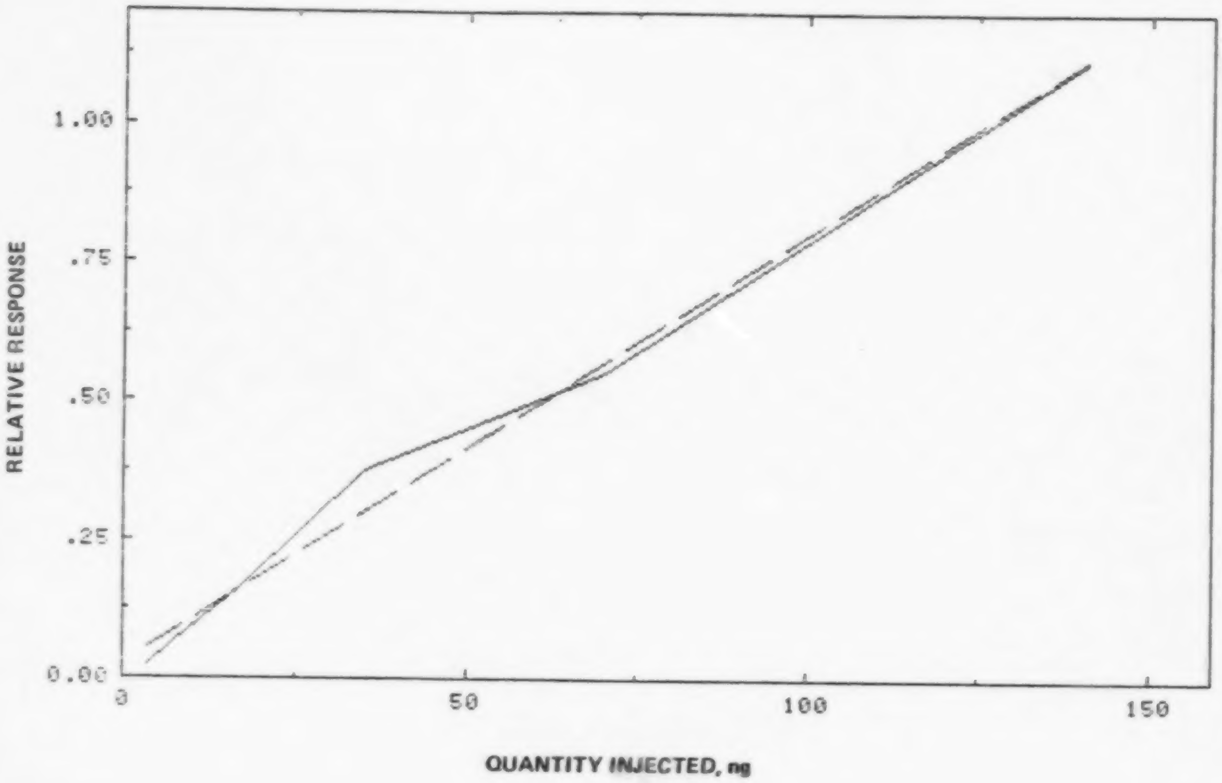
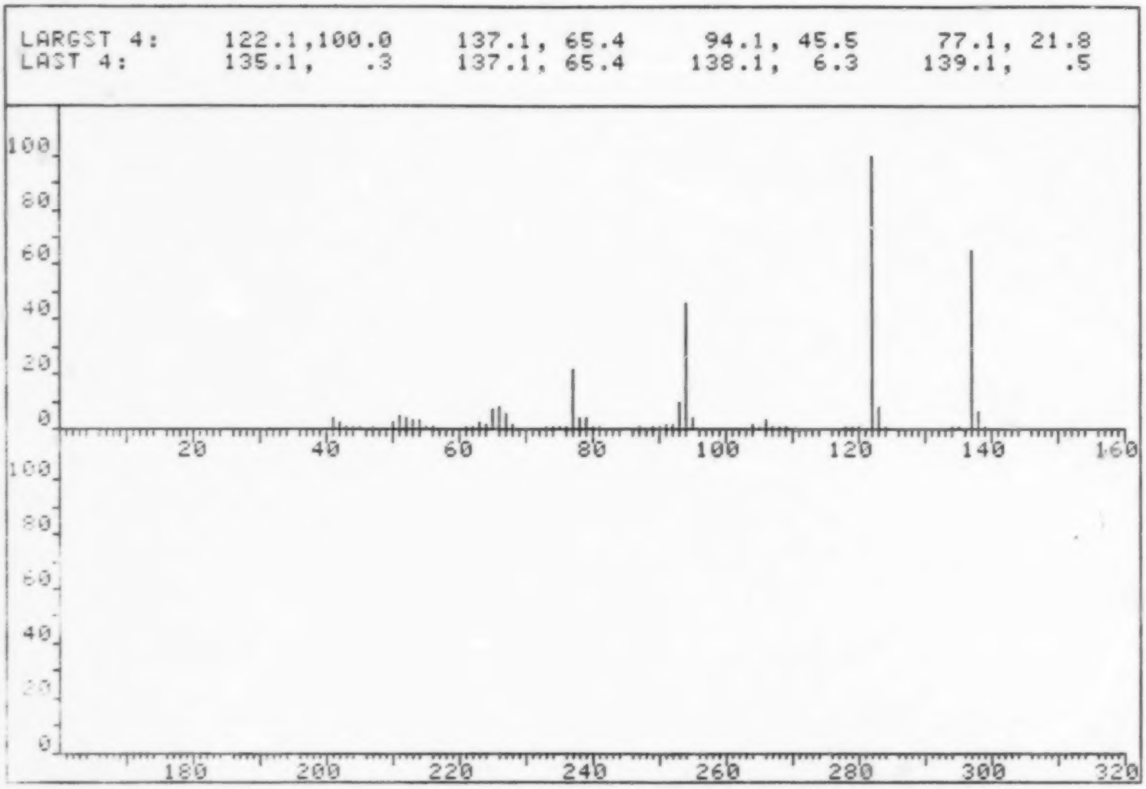


Figure D-95. Calibration curve for the determination of 2-oxepanone by GC/MS.

Range: 18 - 140 ng
Correlation coefficient: 0.996
Slope: 7.7×10^{-3}
y-Intercept: 2.8×10^{-2}



```
LARGST 4:     122.1,100.0     137.1, 65.4      94.1, 45.5      77.1, 21.8
LAST 4:       135.1,    .3    137.1, 65.4     138.1,  6.3     139.1,    .5
```

Figure D-96. Mass spectrum for o-anisidine, 5-methyl-.

TABLE D-42. KEY IONS AND RELATIVE ABUNDANCES FOR o-ANISIDINE, 5-METHYL-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	3.9	63	2.4	78	4.3	106	3.1
42	2.1	64	1.5	79	4.2	107	1.1
50	2.5	65	7.0	80	.9	108	[illegible]
51	4.9	66	7.7	89	.7	120	.4
52	4.3	67	5.7	91	1.5	122	100.0
53	3.3	68	1.5	92	1.9	123	8.5
54	2.9	74	.7	93	10.0	124	.6
55	1.2	75	.6	94	45.5	137	65.4
61	.6	76	1.2	95	3.9	138	6.3
62	1.2	77	21.8	104	1.8		

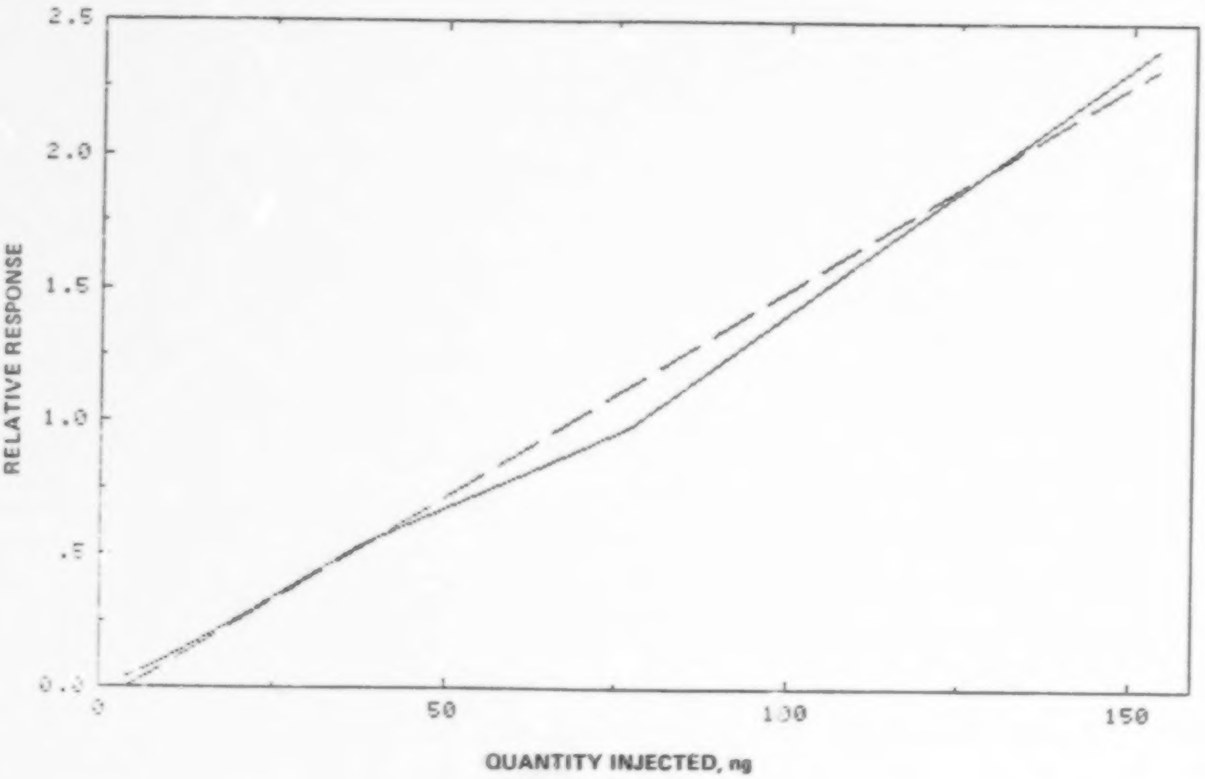


Figure D-97. Calibration curve for the determination of o-anisidine, 5-methyl- by GC/MS.

Range: 19 - 150 ng
Correlation coefficient: 0.996
Slope: 1.6×10^{-2}
y-Intercept: -6.2×10^{-2}

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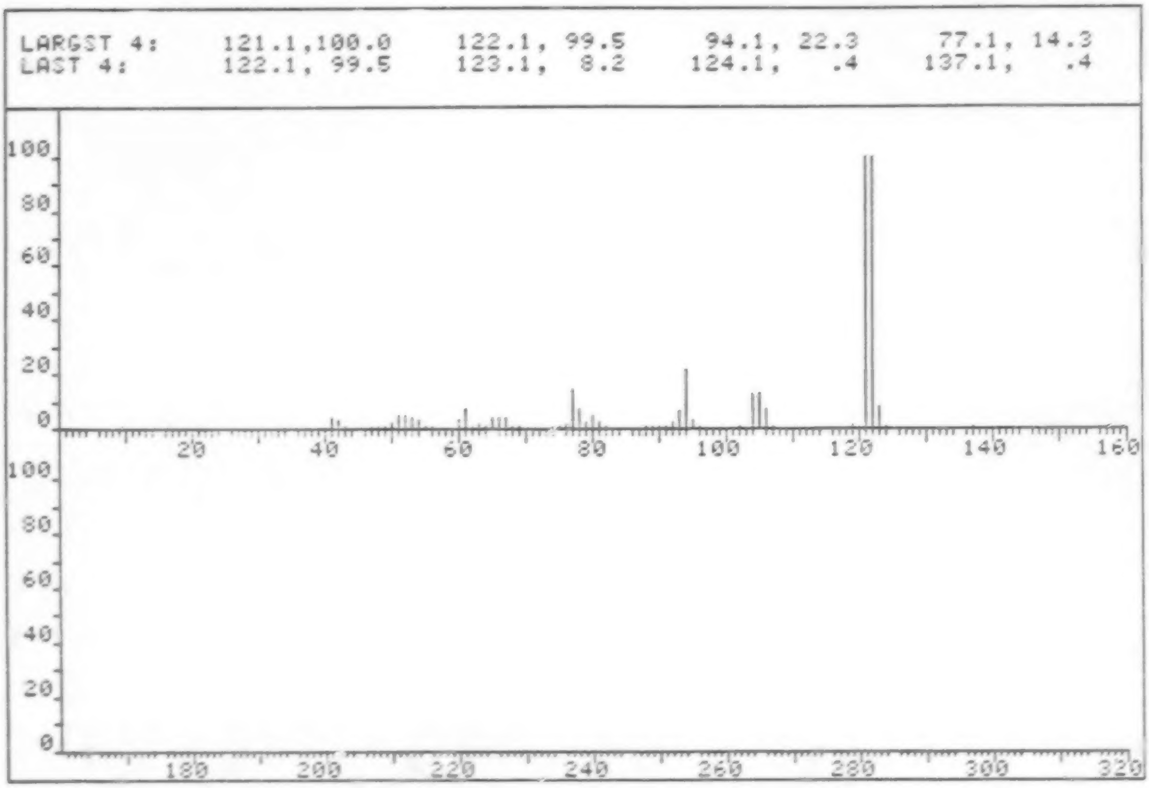


```
LARGST 4:     121.1,100.0     122.1, 99.5      94.1, 22.3      77.1, 14.3
LAST 4:       122.1, 99.5     123.1,  8.2     124.1,   .4     137.1,   .4
```

Figure D-98. Mass spectrum for toluene, 2,4-diamino-.

TABLE D-43. KEY IONS AND RELATIVE ABUNDANCES FOR TOLUENE, 2,4-DIAMINO-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	4.6	61	6.9	77	14.3	102	.6
42	3.3	62	1.2	78	7.1	104	13.3
43	.8	63	1.4	79	2.4	105	12.7
47	.5	64	1.0	80	4.8	106	7.1
48	1.1	65	4.2	81	2.1	107	.9
50	2.7	66	4.5	82	.6	119	1.8
51	5.0	67	4.6	91	.6	121	100.0
52	5.2	68	1.3	92	2.0	122	99.5
53	4.0	69	.5	93	6.4	123	8.2
54	3.7	74	.6	94	22.3		
55	.7	75	.9	95	3.2		
60	2.8	76	1.8	96	.5		

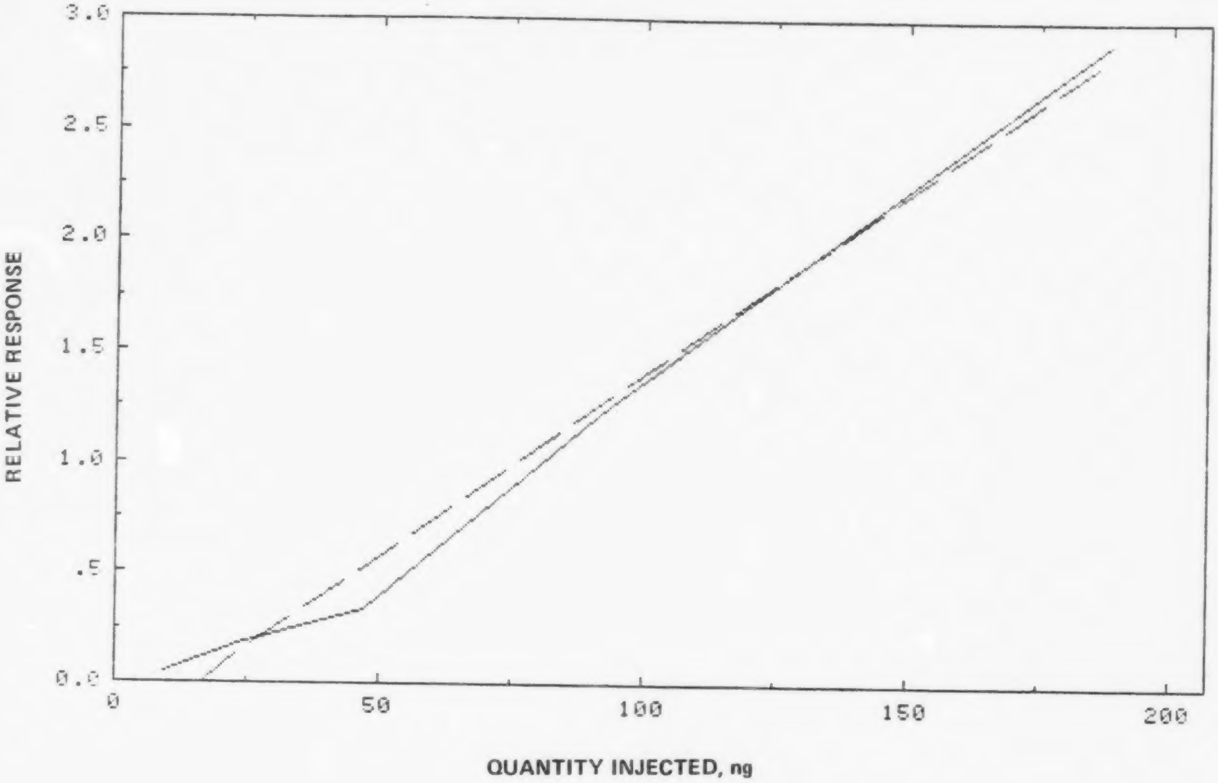
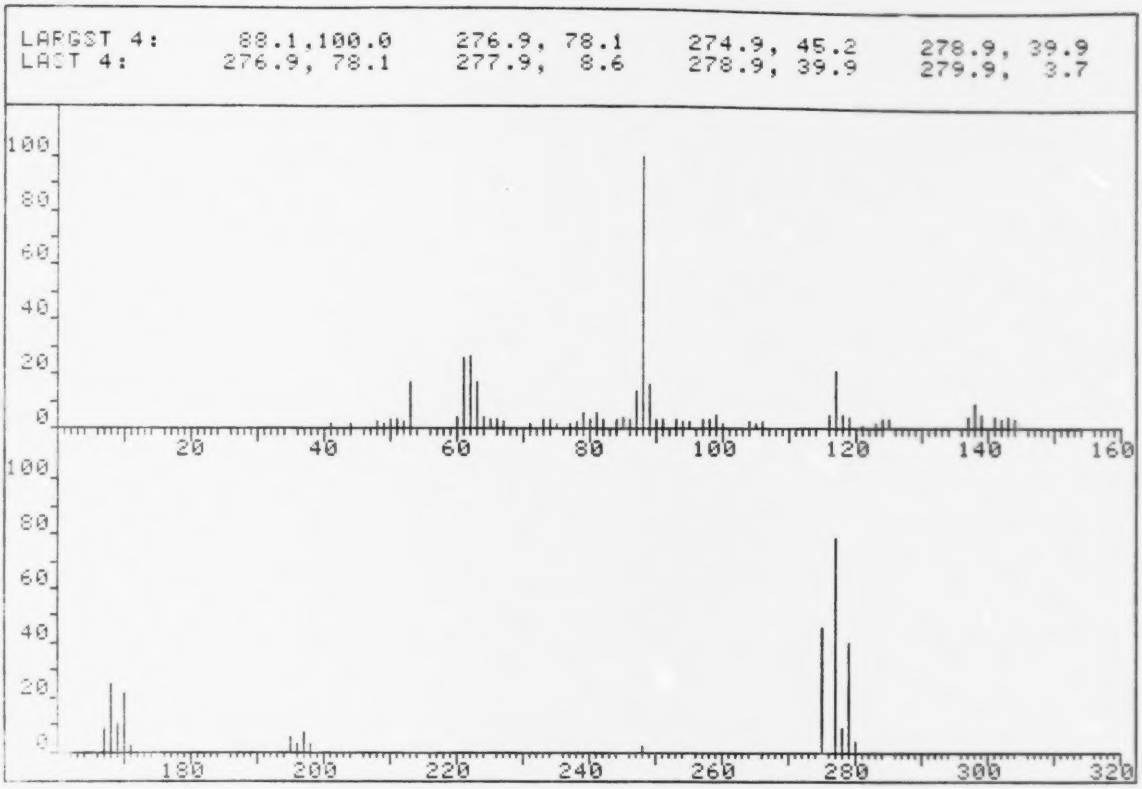


Figure D-99. Calibration curve for the determination of toluene, 2,4-diamino- by GC/MS.

Range: 24 - 190 ng
Correlation coefficient: 0.994
Slope: 1.6×10^{-2}
y-Intercept: -2.4×10^{-1}



```
LARGST 4:    88.1,100.0    276.9, 78.1    274.9, 45.2    278.9, 39.9
LAST 4:     276.9, 78.1    277.9,  8.6    278.9, 39.9    279.9,  3.7
```

Figure D-100. Mass spectrum for benzonitrile, 3,5-dibromo-4-hydroxy-.

TABLE D-44. KEY IONS AND RELATIVE ABUNDANCES FOR BENZONITRILE, 3,5-DIBROMO-4-HYDROXY-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	1.8	75	1.9	98	2.8	143	4.6
44	1.4	77	1.9	99	4.8	144	3.9
48	2.5	78	2.3	100	1.8	167	8.5
49	1.8	79	5.5	104	2.3	168	24.3
50	3.9	80	2.8	105	1.9	169	10.4
51	3.3	81	5.5	106	2.6	170	21.2
52	2.6	82	3.0	116	5.3	171	2.3
53	17.5	84	3.7	117	21.2	195	5.6
60	4.6	85	4.4	118	5.1	196	3.0
61	26.1	86	3.9	119	4.6	197	6.7
62	27.2	87	13.6	121	.7	198	3.2
63	16.6	88	100.0	123	1.9	248	2.3
64	4.6	89	16.0	124	3.9	275	45.1
65	3.5	90	3.2	125	3.2	277	78.1
66	3.9	91	3.5	137	4.2	278	3.6
67	2.3	93	3.3	138	9.2	279	39.9
71	1.8	94	2.1	139	4.8	280	3.7
73	3.3	95	2.3	141	4.1		
74	3.0	97	3.0	142	3.7		

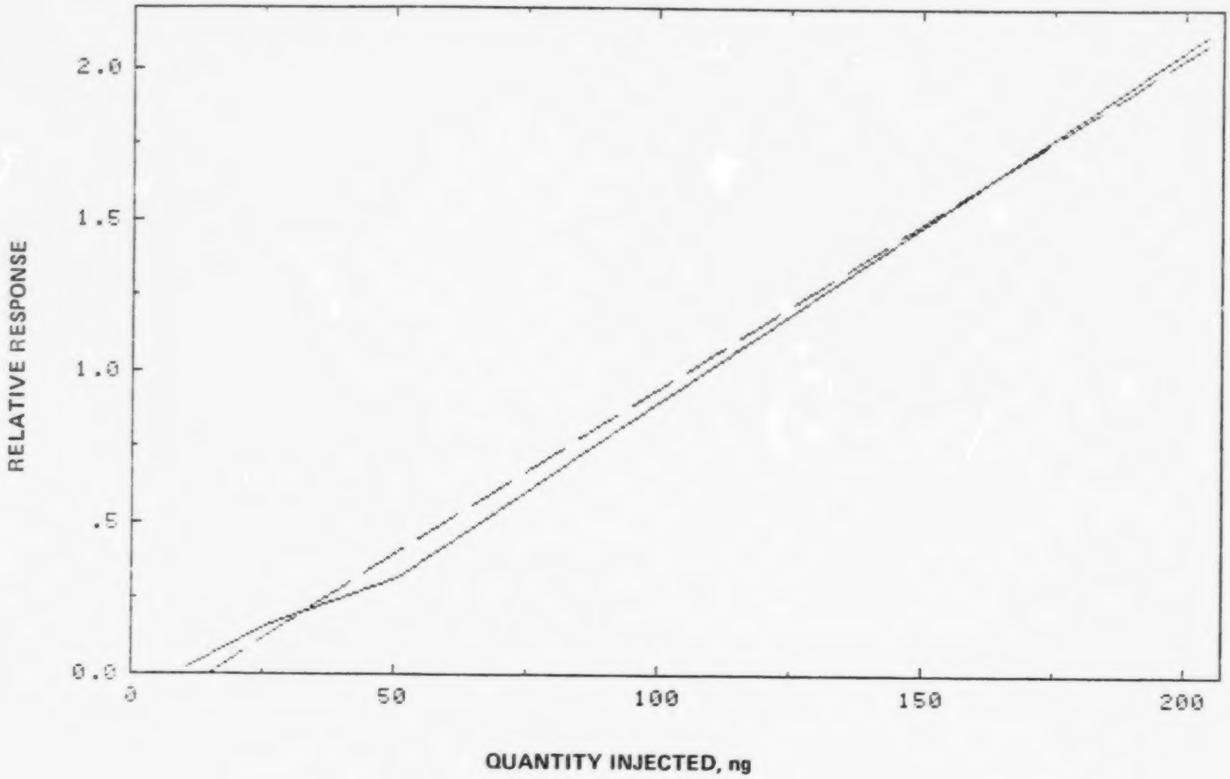


Figure D-101. Calibration curve for the determination of benzonitrile, 3,5-dibromo-4-hydroxy- by GC/MS.

Range: 26 - 200 ng
Correlation coefficient: 0.997
Slope: 1.1×10^{-2}
y-Intercept: -1.5×10^{-1}

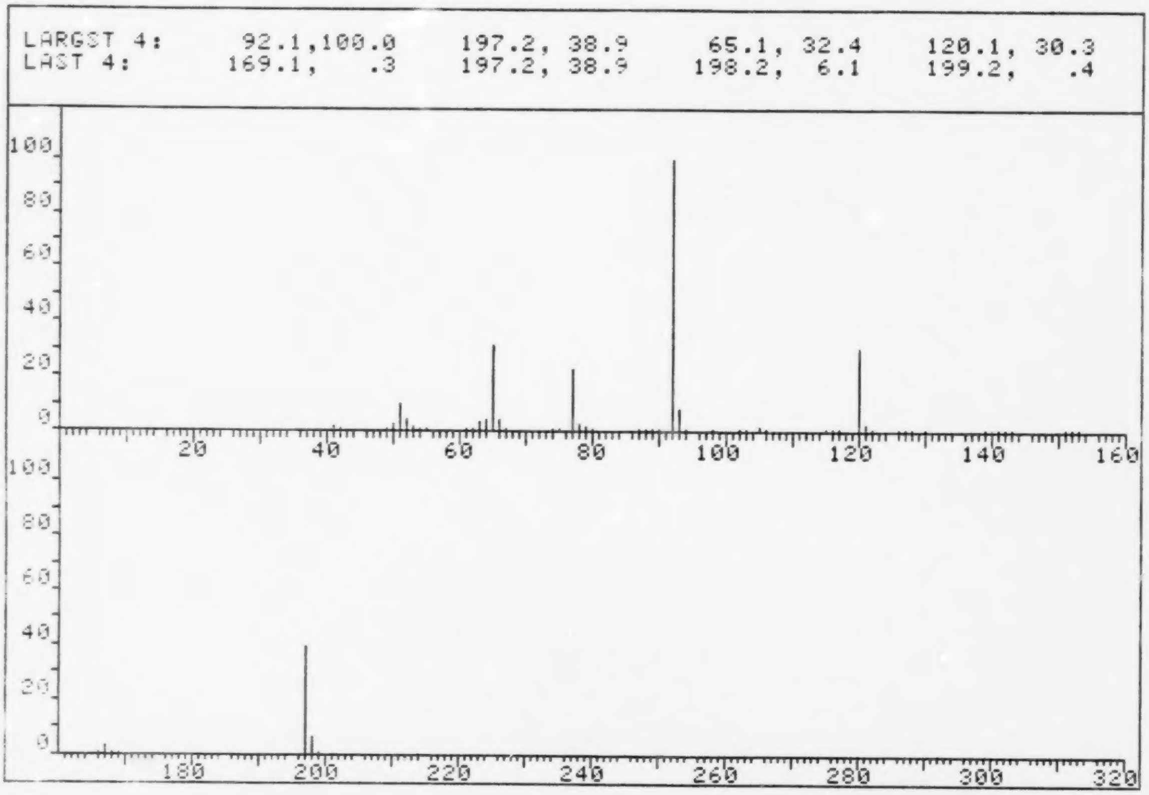


Figure D-102. Mass spectrum for aniline, p-(phenylazo)-.

TABLE D-45. KEY IONS AND RELATIVE ABUNDANCES FOR ANILINE, p-(PHENYLAZO)-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	1.7	64	3.9	92	100.0	151	.7
42	.8	65	32.4	93	8.1	152	.7
50	2.3	66	4.2	98	.7	166	.5
51	9.6	74	.7	105	2.0	167	2.5
52	4.4	75	.8	106	.6	168	.8
53	1.5	77	22.7	115	1.2	197	38.9
54	1.0	78	2.5	120	30.3	198	6.1
62	1.0	79	1.4	121	2.1		
63	3.1	90	.7	141	1.0		

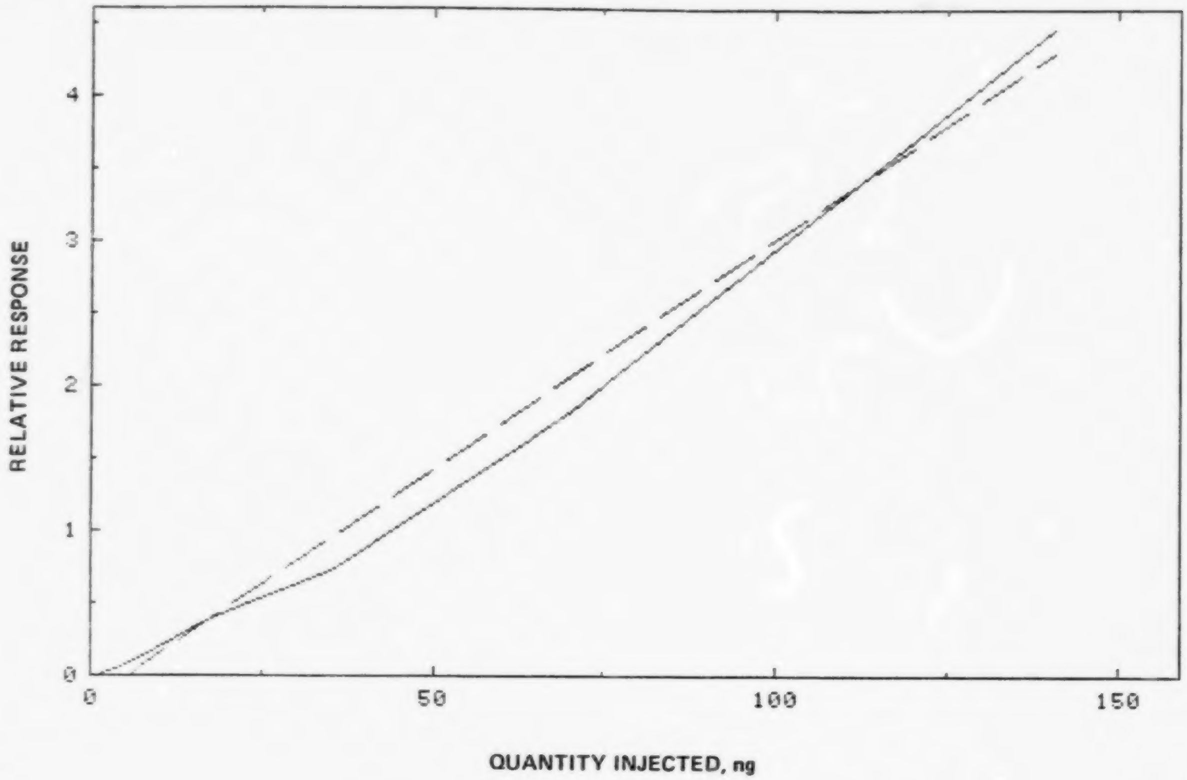


Figure D-103. Calibration curve for the determination of aniline, p-(phenylazo)- by GC/MS.

Range: 18 - 140 ng
Correlation coefficient: 0.995
Slope: 3.2×10^{-2}
y-Intercept: -1.7×10^{-1}

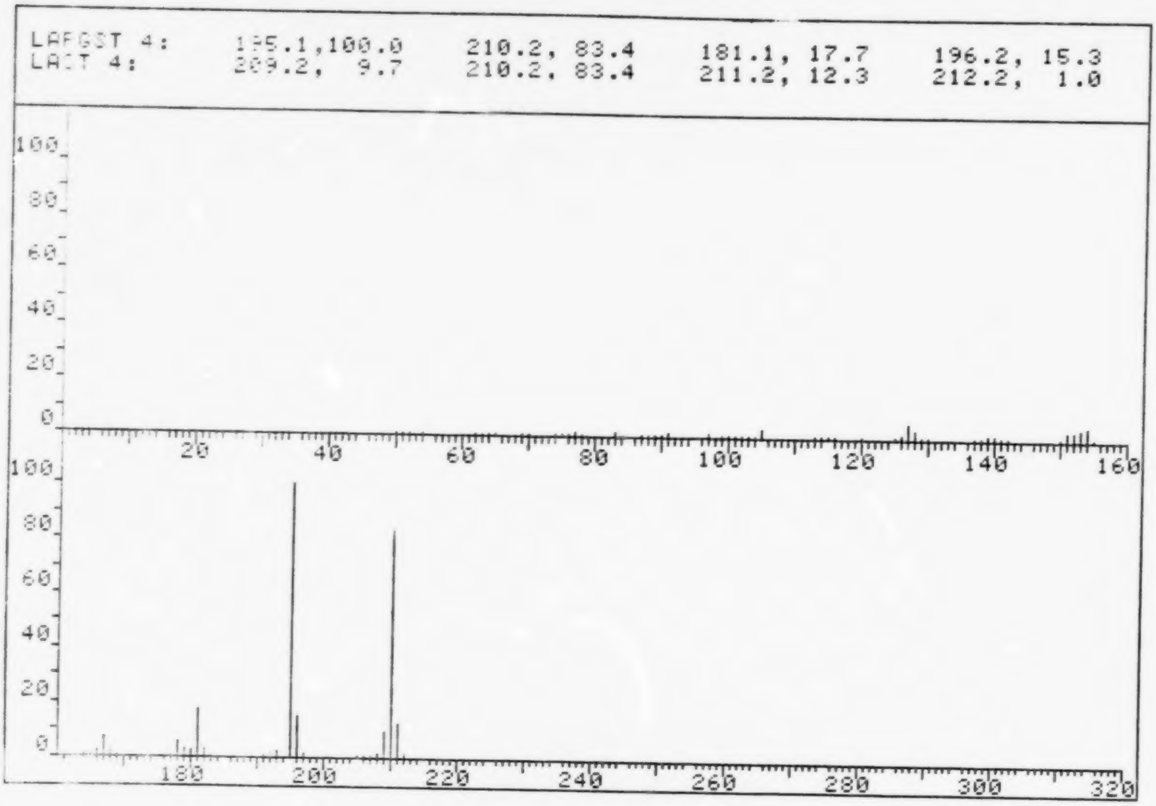


Figure D-104. Mass spectrum for 3-amino-9-ethyl carbazole.

TABLE D-46. KEY IONS AND RELATIVE ABUNDANCES FOR 3-AMINO-9-ETHYL CARBAZOLE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
50	.5	100	.6	141	1.1	182	3.3
51	.8	101	1.2	151	3.0	191	.7
52	.6	102	.7	152	3.0	192	1.5
63	1.1	103	.6	153	3.9	193	2.1
64	.6	104	.6	154	4.7	195	100.0
74	.6	105	3.3	155	1.0	196	15.3
75	1.2	113	.5	164	.8	197	1.1
76	1.0	114	.5	165	.5	207	.8
77	2.0	115	.9	166	2.4	208	1.3
78	.6	125	.6	167	6.6	209	9.7
83	1.4	126	1.7	168	2.2	210	83.4
88	.7	127	5.7	177	.8	211	12.3
89	.6	128	3.0	178	5.0	212	1.0
90	1.1	129	.6	179	3.0		
91	1.7	139	1.5	180	1.8		
97	1.7	140	2.0	181	17.7		

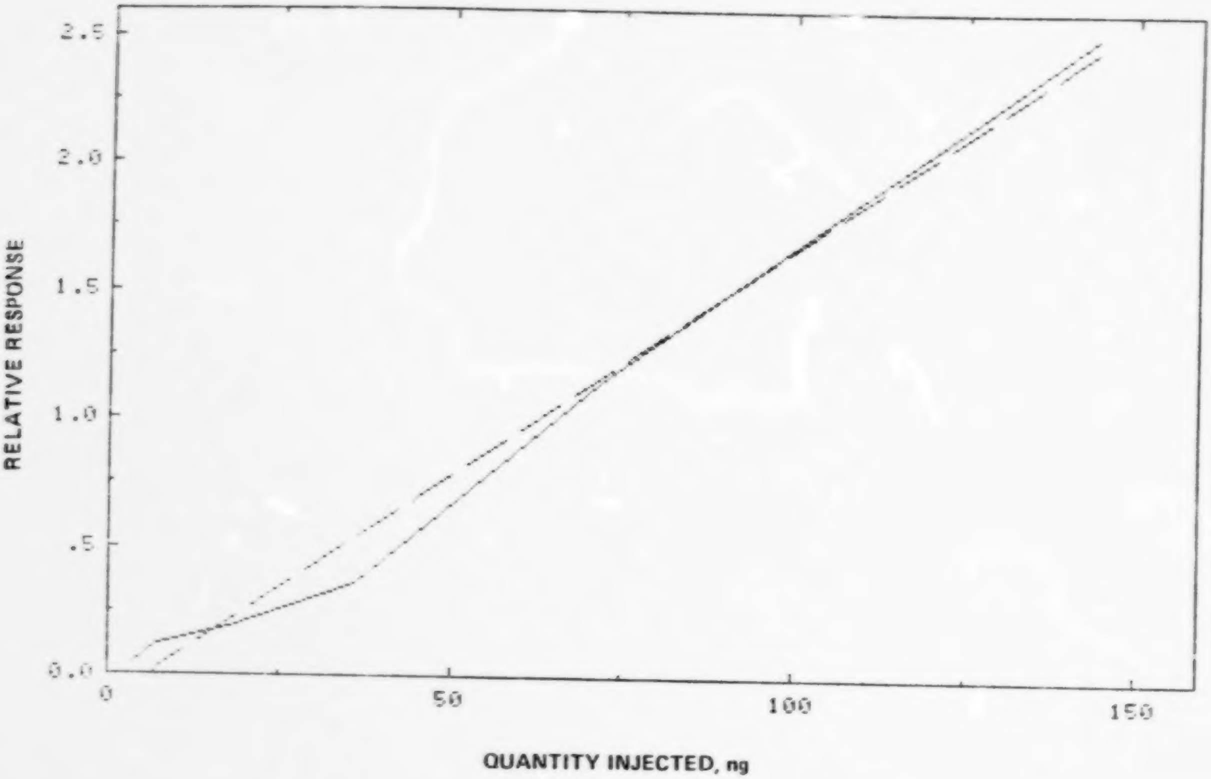
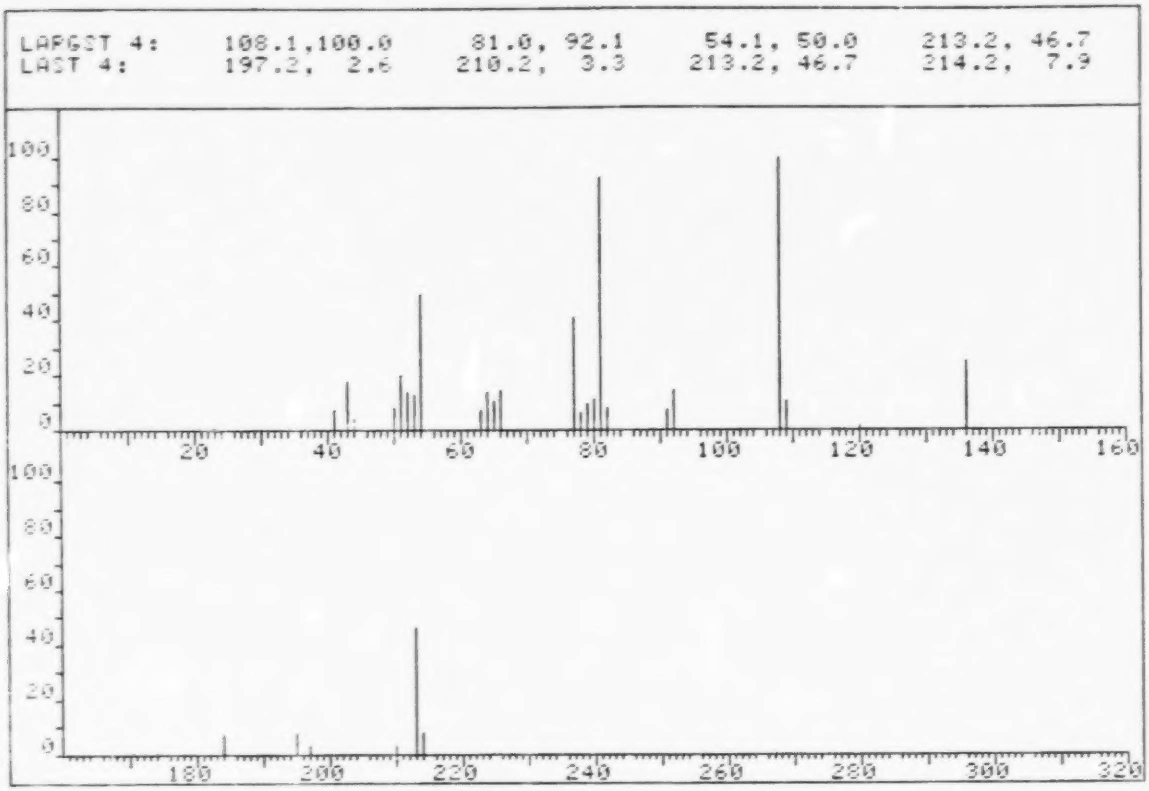


Figure D-105. Calibration curve for the determination of 3-amino-9-ethyl carbazole by GC/MS.

Range: 18 - 140 ng
Correlation coefficient: 0.994
Slope: 1.8×10^{-2}
y-Intercept: -1.0×10^{-1}



```
LARGST 4:     108.1,100.0     81.0, 92.1     54.1, 50.0     213.2, 46.7
LAST 4:       197.2,  2.6    210.2,  3.3    213.2, 46.7     214.2,  7.9
```

Figure D-106. Mass spectrum for pyridine, 2,6-diamino-3-(phenylazo)-, monohydrochloride.

TABLE D-47. KEY IONS AND RELATIVE ABUNDANCES FOR PYRIDINE, 2,6-DIAMINO-3-(PHENYLAZO)-, MONOHYDROCHLORIDE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	7.2	63	7.2	81	92.1	184	6.6
43	17.8	64	13.8	82	8.6	195	7.2
44	4.6	65	10.5	91	7.2	197	2.6
50	7.9	66	14.5	92	14.5	210	3.3
51	20.4	77	40.8	108	100.0	213	46.7
52	13.8	78	6.6	109	10.5	214	7.9
53	13.2	79	9.9	120	1.3		
54	50.0	80	11.2	136	25.0		

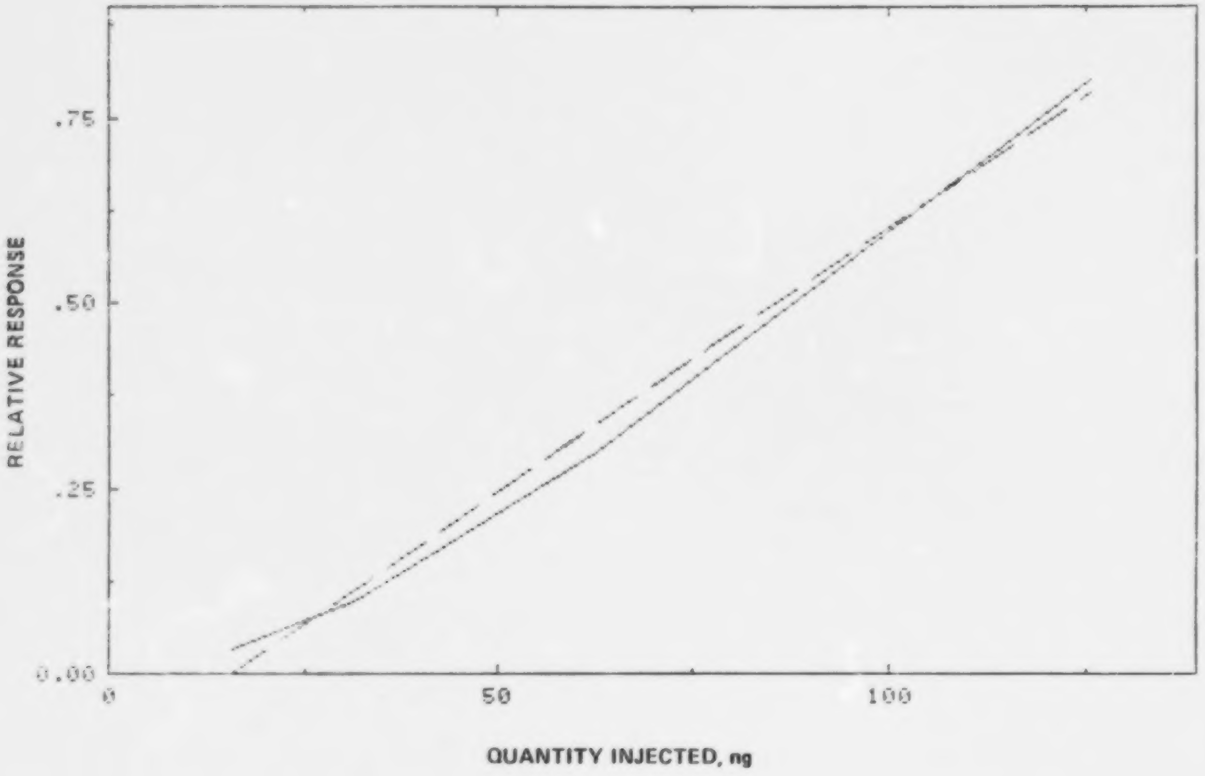


Figure D-107. Calibration curve for the determination of pyridine, 2,6-diamino-3-(phenylazo)-, monohydrochloride by GC/MS.

Range: 31 - 130 ng
Correlation coefficient: 0.996
Slope: 7.1×10^{-3}
y-Intercept: -1.1×10^{-1}

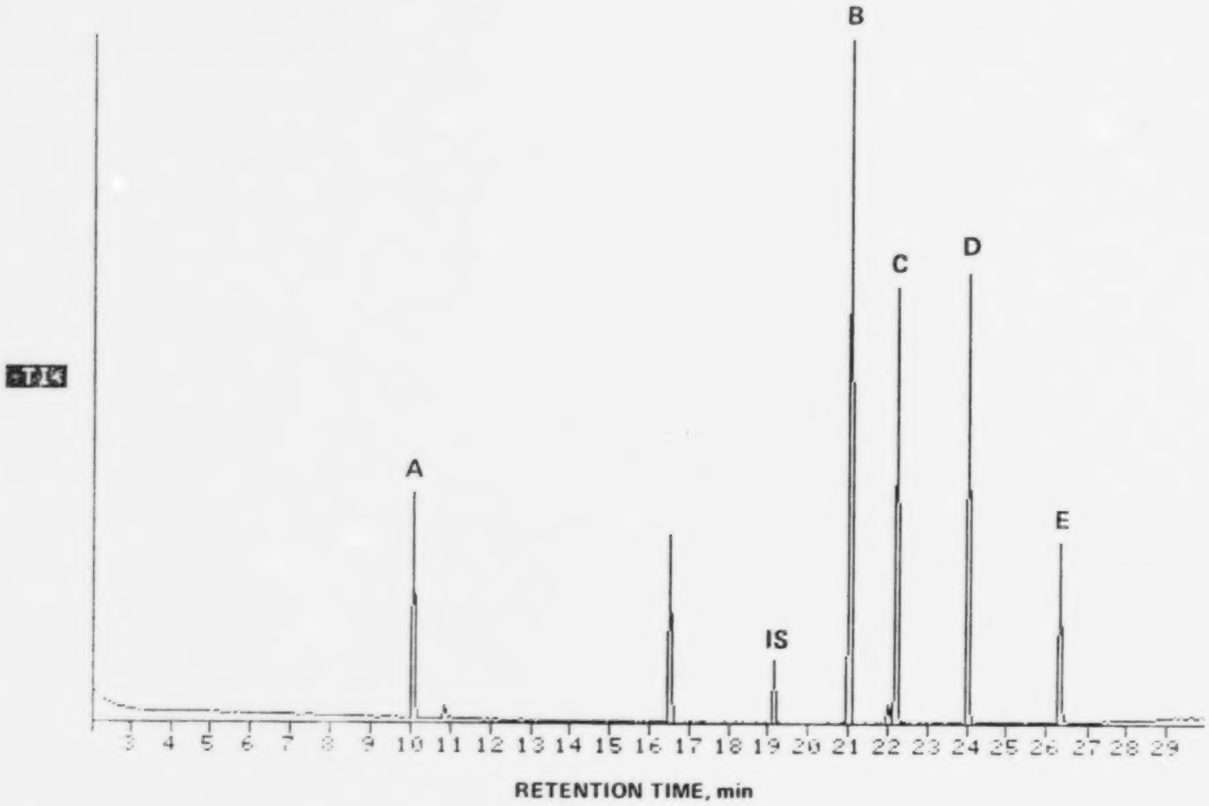
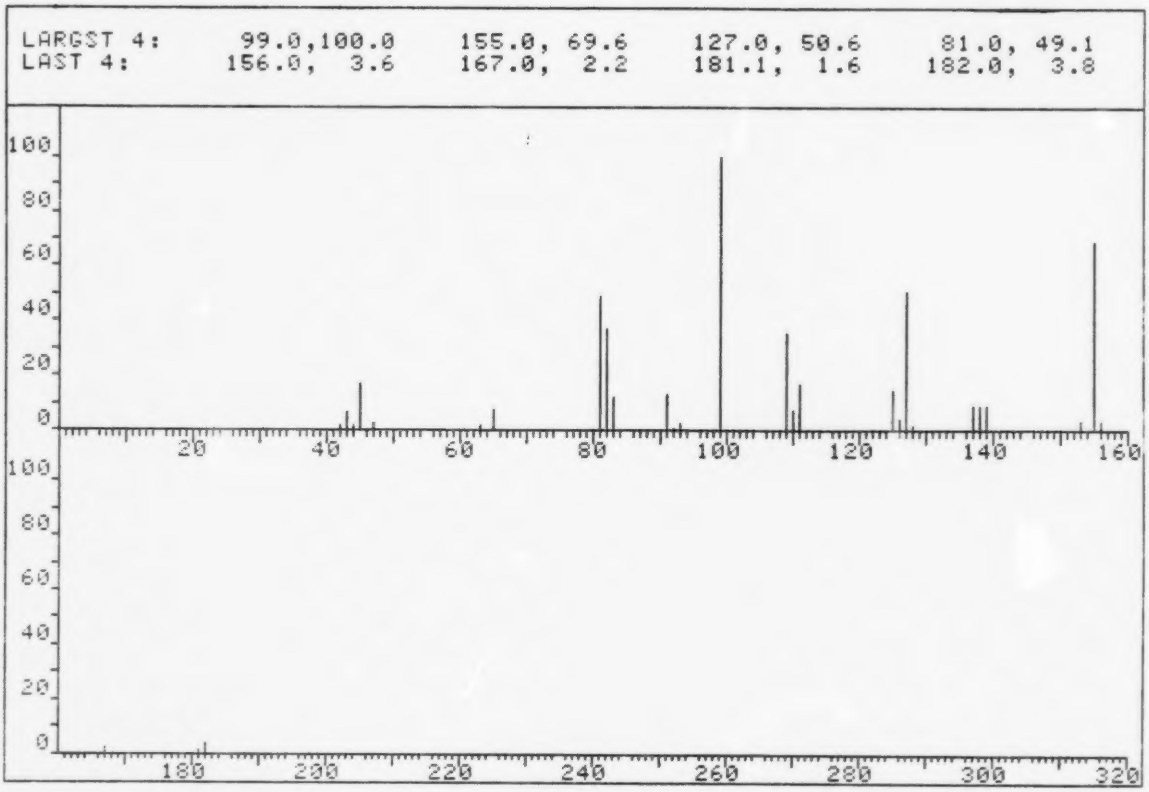


Figure D-108. Chromatogram by GC/MS for

(A) tetraethylpyrophosphate(t_R = 10.1 min),
(B) succinic acid, mercapto-, diethyl ester, *S*-ester with *O*,*O*-dimethyl phosphorodithioate(t_R = 21.1 min),
(C) crotonic acid, 3-hydroxy-, alpha-methylbenzyl ester, dimethyl phosphate, (*E*)- (t_R = 22.2 min),
(D) phosphorodithioic acid, *S*,*S*'-methylene *O*,*O*,*O*',*O*'-tetraethyl ester(t_R = 24.0 min), and
(E) phosphorodithioic acid, *O*,*O*-dimethyl ester, *S*-ester with 3-(mercaptomethyl)-1,2,3-benzotriazin-4(3*H*)-one(t_R = 26.4 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal



```
LARGST 4:     99.0,100.0     155.0, 69.6     127.0, 50.6      81.0, 49.1
LAST 4:      156.0,   3.6    167.0,  2.2     181.1,  1.6     182.0,  3.8
```

Figure D-109. Mass spectrum for tetraethylpyrophosphate.

TABLE D-48. KEY IONS AND RELATIVE ABUNDANCES FOR TETRAETHYLPYROPHOSPHATE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
42	1.5	82	37.3	111	16.5	153	2.9
43	6.3	83	12.4	125	14.9	155	69.6
44	1.8	91	13.2	126	4.4	156	3.6
45	17.2	92	1.1	127	50.6	167	2.2
47	2.2	93	2.6	128	1.6	181	1.6
63	1.4	99	100.0	137	9.2	182	3.8
65	7.1	109	35.1	138	8.9		
81	49.1	110	7.6	139	8.7		

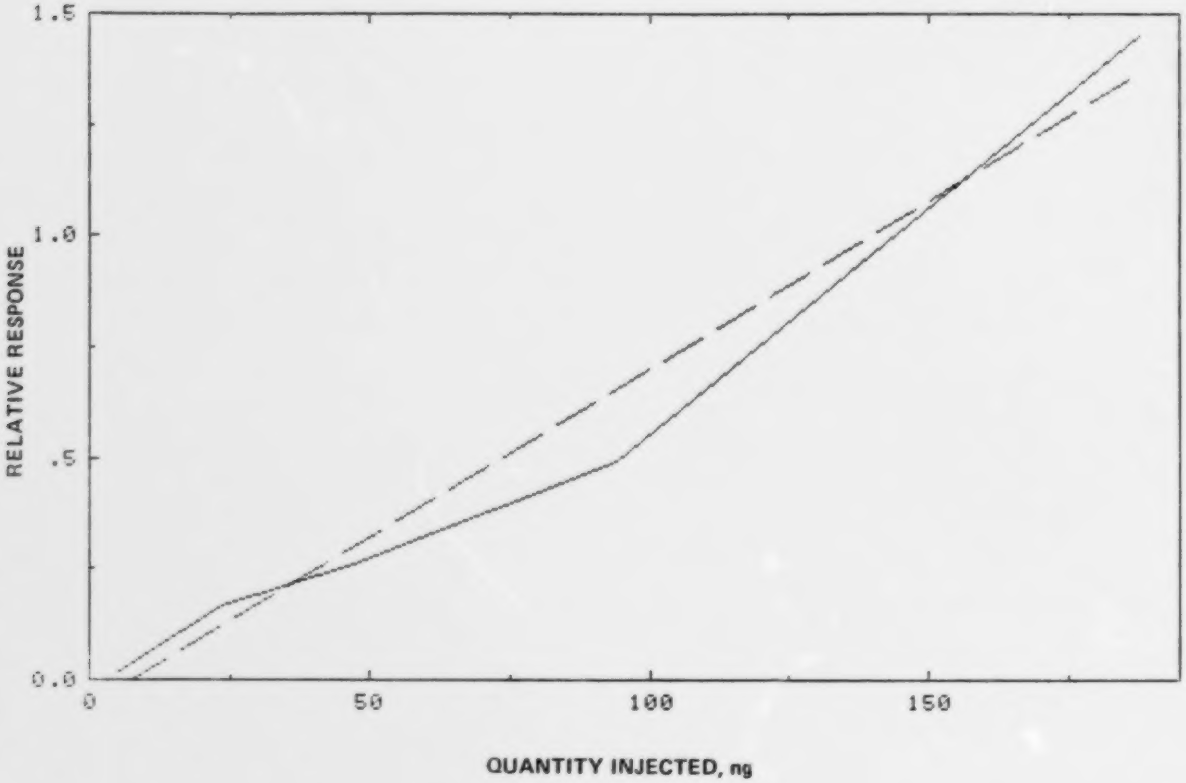
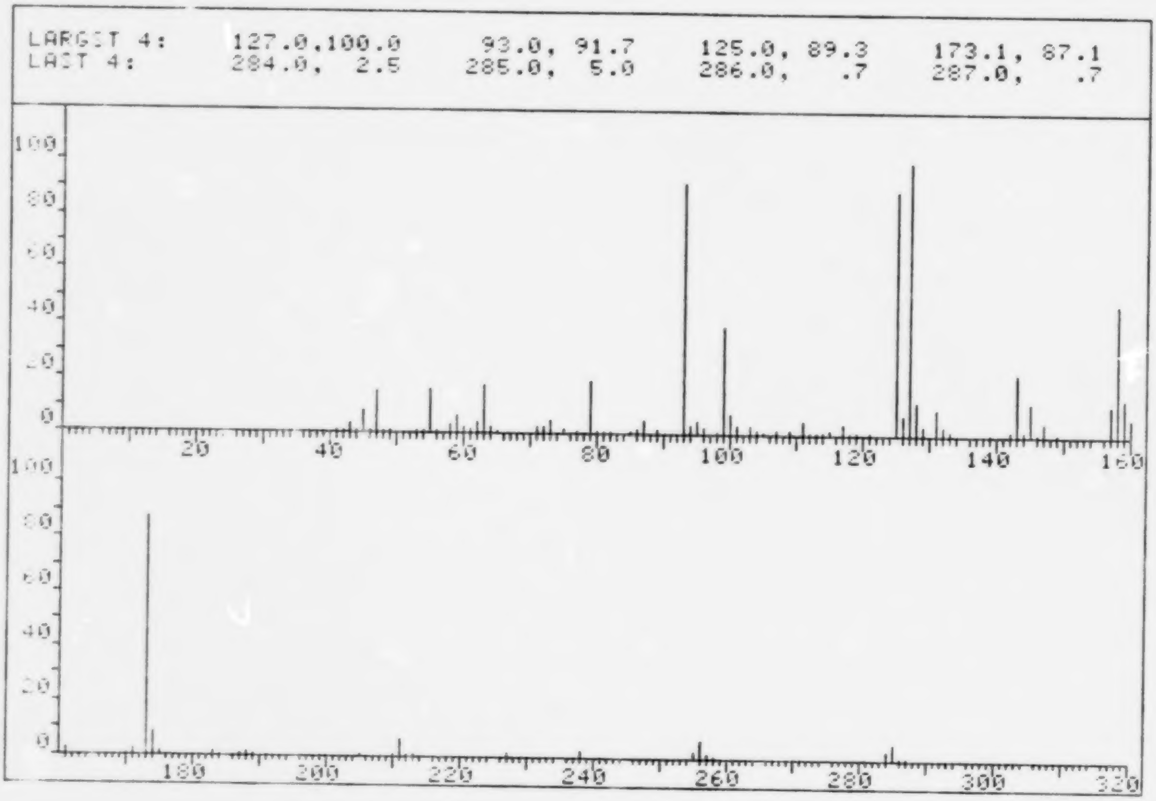


Figure D-110. Calibration curve for the determination of tetraethylpyrophosphate by GC/MS.

Range: 24 - 190 ng
Correlation coefficient: 0.986
Slope: 7.6×10^{-3}
y-Intercept: -5.5×10^{-2}



```
LARGST 4:     127.0,100.0      93.0, 91.7     125.0, 89.3     173.1, 87.1
LAST 4:       284.0,   2.5    285.0,  5.0     286.0,    .7    287.0,    .7
```

Figure D-111. Mass spectrum for succinic acid, mercapto-, diethyl ester, S-ester with O,O-dimethyl phosphorodithioate.

TABLE D-49. KEY IONS AND RELATIVE ABUNDANCES FOR SUCCINIC ACID, MERCAPTO-, DIETHYL ESTER, S-ESTER WITH O,O-DIMETHYL PHOSPHORODITHIOATE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	.7	80	1.0	118	.6	173	87.1
42	.7	81	1.0	119	.6	174	8.4
43	3.1	82	1.0	121	.9	175	1.5
45	7.9	85	.9	125	89.3	183	1.0
46	1.1	86	1.8	126	7.3	184	1.1
47	15.0	87	5.3	127	100.0	187	.6
48	.9	89	1.6	128	12.2	188	1.0
49	1.0	93	91.7	129	2.8	189	.9
53	1.1	94	3.2	131	9.7	205	.7
54	1.2	95	4.9	132	3.1	210	.7
55	15.8	96	2.6	133	1.5	211	6.1
56	.8	98	.9	139	1.0	212	.7
58	3.0	99	39.2	142	1.7	213	.6
59	6.3	100	6.9	143	22.6	227	1.5
60	2.2	101	3.4	144	1.9	238	2.1
61	1.8	103	3.8	145	11.9	255	2.3
62	4.5	104	1.4	146	1.2	256	6.3
63	18.0	105	1.0	147	4.7	257	1.5
64	2.7	107	1.8	149	.7	258	.9
65	1.0	109	1.4	157	11.3	284	2.5
71	2.8	111	5.1	158	48.5	285	5.0
72	2.3	112	.8	159	13.7	286	.7
73	5.2	113	1.1	160	6.4	287	.7
75	1.7	114	1.1	161	2.0		
77	1.1	115	1.5	170	.6		
79	19.8	117	4.0	171	2.2		

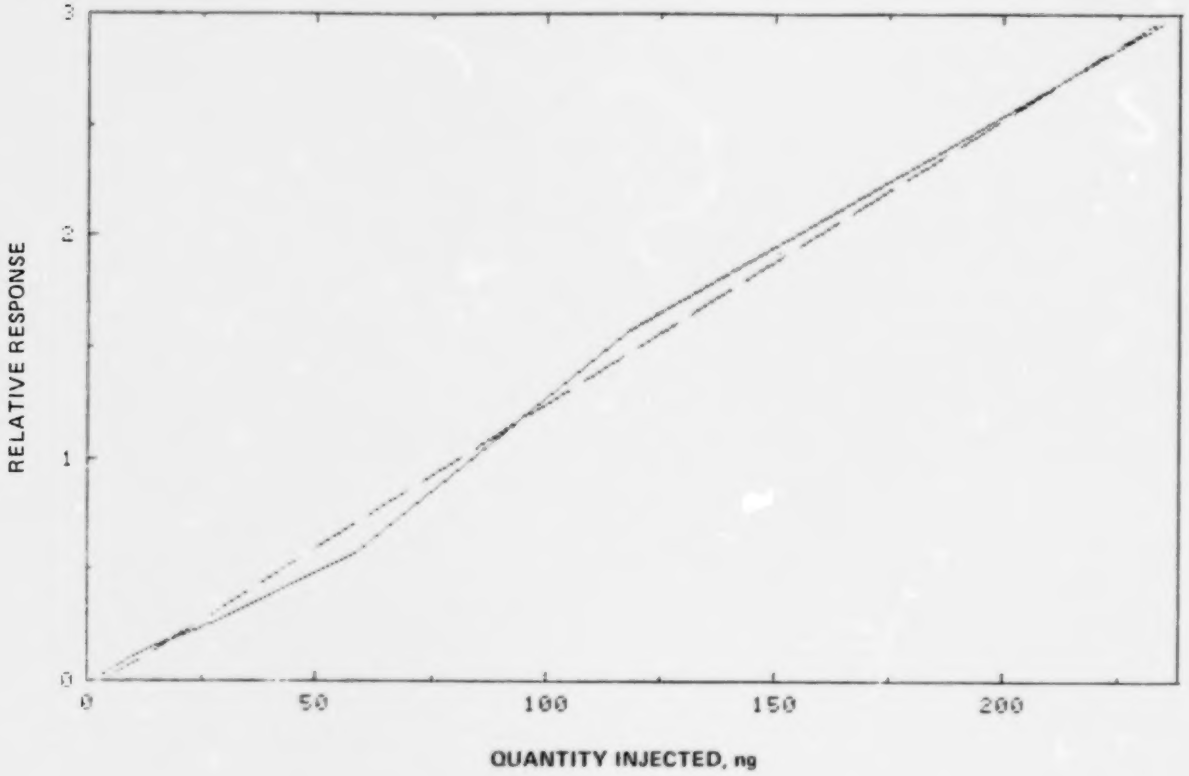
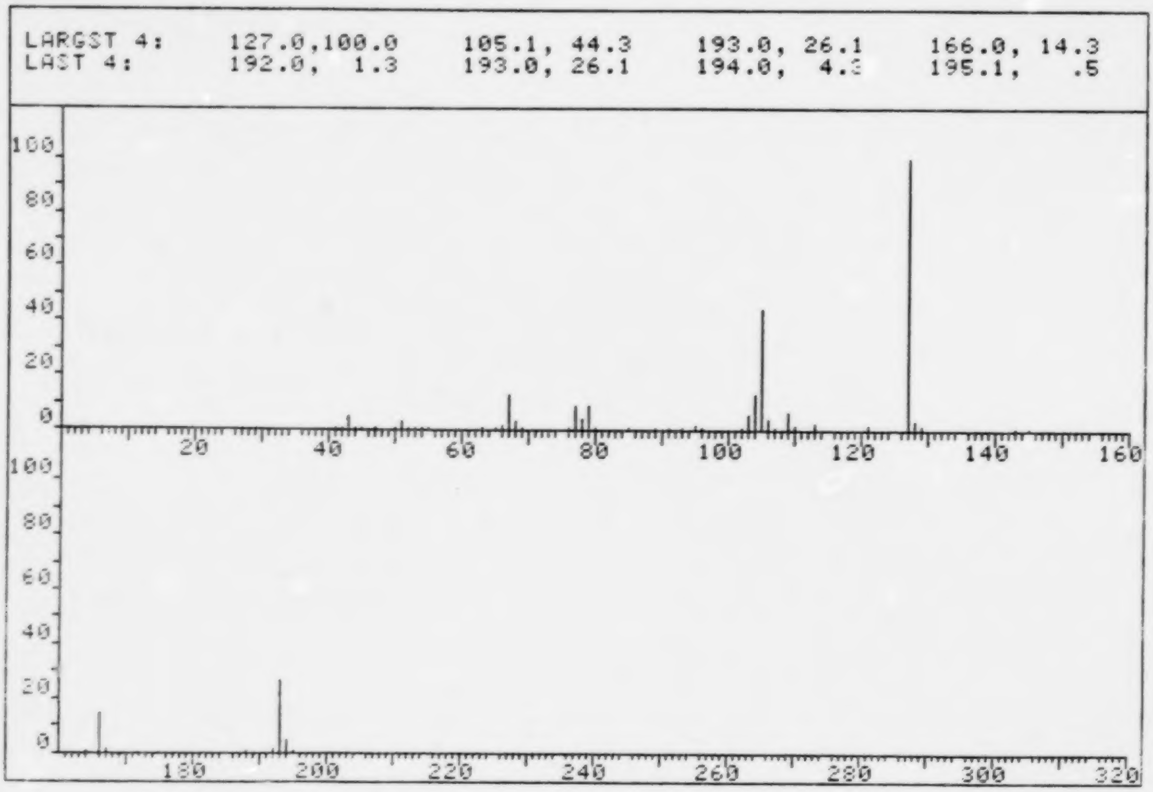


Figure D-112. Calibration curve for the determination of succinic acid, mercapto-, diethyl ester, S-ester with O,O-dimethyl phosphorodithioate by GC/MS.

Range: 12 - 250 ng
Correlation coefficient: 0.998
Slope: 1.3×10^{-2}
y-Intercept: -4.9×10^{-2}



```
LARGST 4:     127.0,100.0     105.1, 44.3     193.0, 26.1     166.0, 14.3
LAST 4:       192.0,  1.3     193.0, 26.1     194.0,  4.3     195.1,   .5
```

Figure D-113. Mass spectrum for crotonic acid, 3-hydroxy-, alpha-methylbenzyl ester, dimethyl phosphate, (*E*)- .

TABLE D-50. KEY IONS AND RELATIVE ABUNDANCES FOR CROTONIC ACID, 3-HYDROXY-, *ALPHA*-METHYLBENZYL ESTER, DIMETHYL PHOSPHATE, (*E*)-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	.5	66	1.4	93	1.2	121	1.3
43	4.9	67	12.9	95	1.4	127	100.0
47	1.0	68	3.1	96	1.2	128	3.0
50	.8	69	1.2	102	.6	129	2.0
51	3.3	76	.5	103	5.5	145	.9
52	.9	77	9.4	104	13.1	166	14.3
53	.5	78	3.9	105	44.3	167	1.0
54	1.3	79	9.1	106	4.2	192	1.3
55	.5	80	.7	109	6.6	193	26.1
63	1.0	85	.7	110	1.0	194	4.3
65	.8	91	.6	113	2.6	195	.5

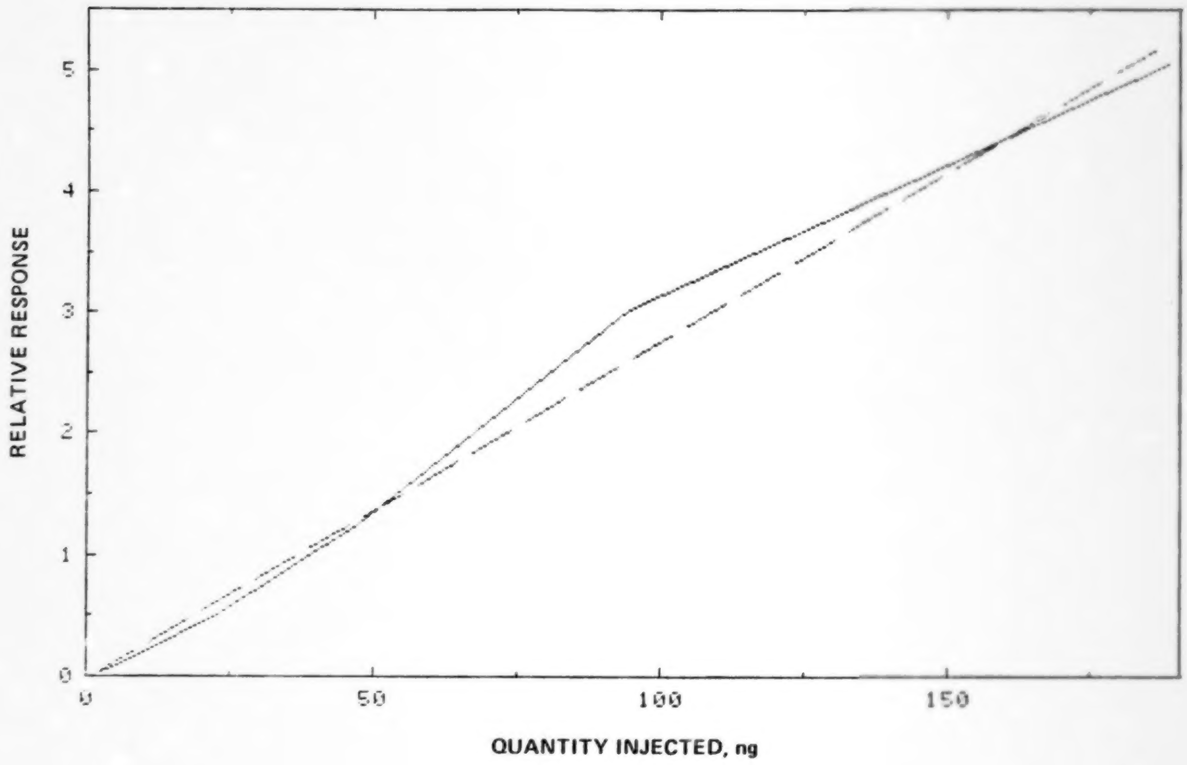


Figure D-114. Calibration curve for the determination of crotonic acid, 3-hydroxy-, alpha-methylbenzyl ester, dimethyl phosphate, (E)- by GC/MS.

Range: 2.4 - 190 ng
Correlation coefficient: 0.995
Slope: 2.8×10^{-2}
y-Intercept: -2.9×10^{-2}

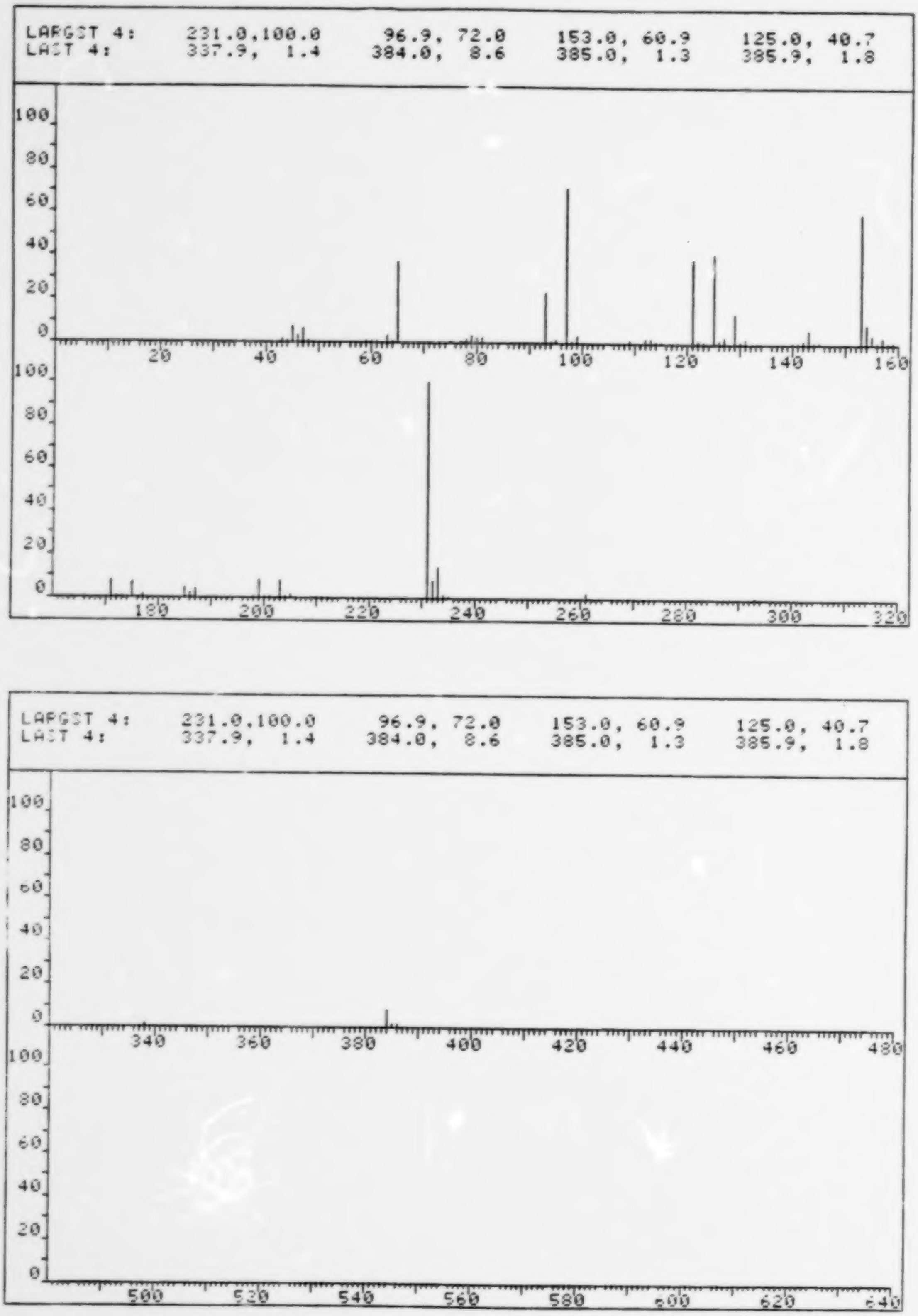


Figure D-115. Mass spectrum for phosphorodithioic acid, S,S'-methylene O,O,O',O'-tetraethyl ester.

TABLE D-51. KEY IONS AND RELATIVE ABUNDANCES FOR PHOSPHORODITHIOIC ACID, S,S-METHYLENE O,O,O',O'-TETRAETHYL ESTER

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
43	1.5	95	1.7	141	1.1	187	3.6
45	6.9	97	72.0	142	2.0	198	.8
46	3.2	98	.9	143	6.2	199	7.6
47	6.3	99	3.4	144	.9	200	.9
48	1.1	109	1.7	145	.7	201	.8
59	.8	112	2.6	153	60.9	203	8.1
60	1.0	113	2.5	154	9.1	204	.7
61	1.1	114	1.1	155	4.6	205	1.3
63	3.6	115	.7	156	.8	231	100.0
64	1.2	121	38.6	157	3.8	232	7.9
65	37.1	122	2.0	158	.9	233	14.4
75	.9	123	.7	159	1.2	234	1.3
77	.8	125	40.7	171	7.3	235	.8
78	1.5	126	2.0	172	.7	261	2.0
79	3.2	127	2.9	173	.9	293	.7
80	2.4	129	14.2	175	7.2	338	1.4
81	2.6	130	.7	177	1.0	384	8.6
93	23.2	131	2.3	185	5.0	385	1.3
94	.7	140	.8	186	2.2	386	1.8

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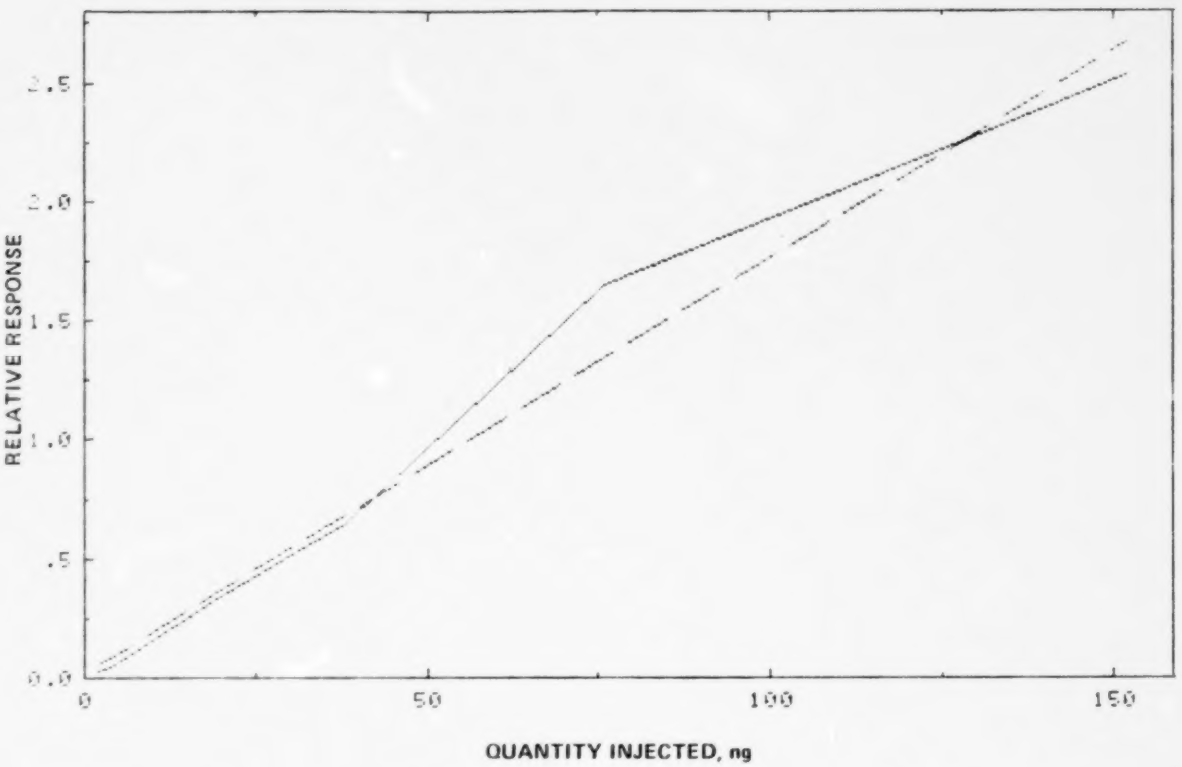


Figure D-116. Calibration curve for the determination of phosphorodithioic acid, S,S'-methylene O,O,O',O'-tetraethyl ester by GC/MS.

Range: 7.6 - 150 ng
Correlation coefficient: 0.989
Slope: 1.7 x 10^{-2}
y-Intercept: 1.9 x 10^{-2}

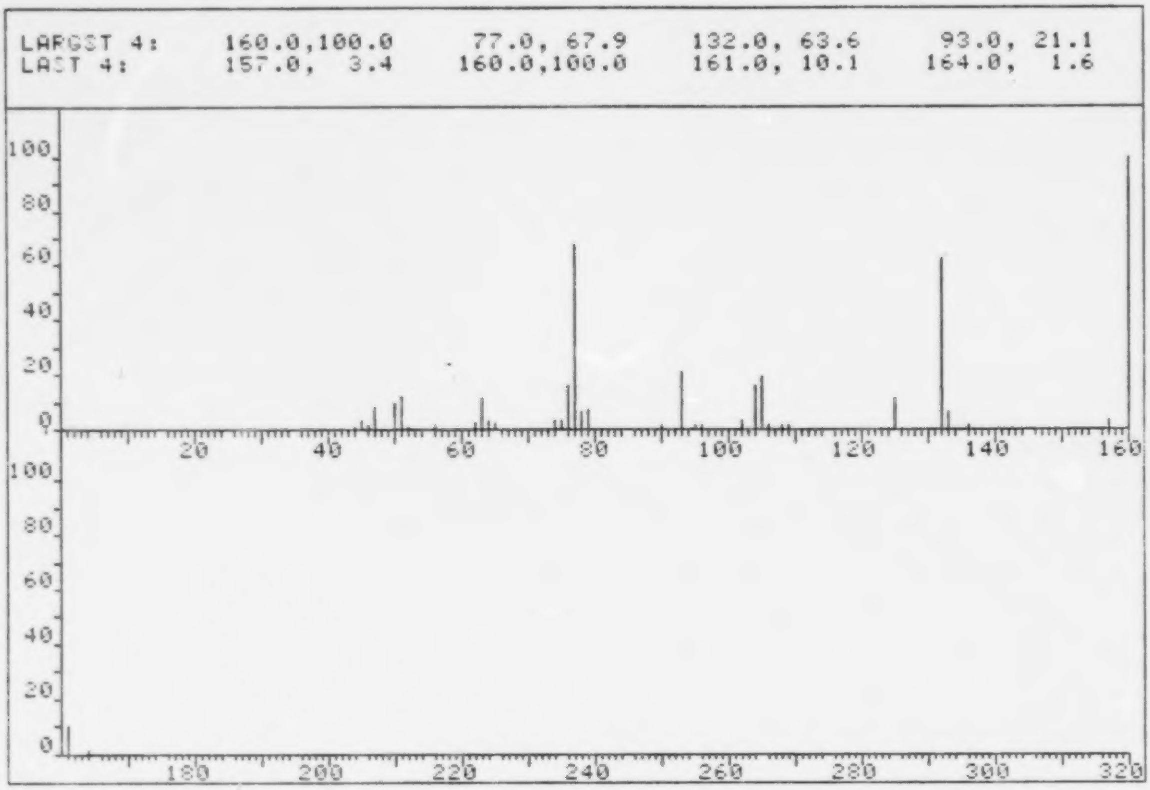


Figure D-117. Mass spectrum for phosphorodithioic acid, *O*,*O*-dimethyl ester, *S*-ester with 3-(mercaptomethyl)-1,2,3-benzotriazin-4(3*H*)-one.

TABLE D-52. KEY IONS AND RELATIVE ABUNDANCES FOR PHOSPHORODITHIOIC ACID, O,O-DIMETHYL ESTER, S-ESTER WITH 3-(MERCAPTOMETHYL)-1,2,3-BENZOTRIAZIN-4(3H)-ONE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
44	.5	63	11.2	90	1.7	109	1.9
45	3.5	64	3.1	93	21.1	125	11.2
46	1.5	65	2.2	95	2.0	132	63.6
47	8.6	74	3.0	96	1.7	133	6.4
50	10.1	75	3.1	102	3.7	136	1.6
51	11.9	76	15.9	104	15.7	157	3.4
52	1.2	77	67.9	105	19.1	160	100.0
56	2.0	78	6.2	106	1.6	161	10.1
52	2.7	79	7.1	108	1.4	164	1.6

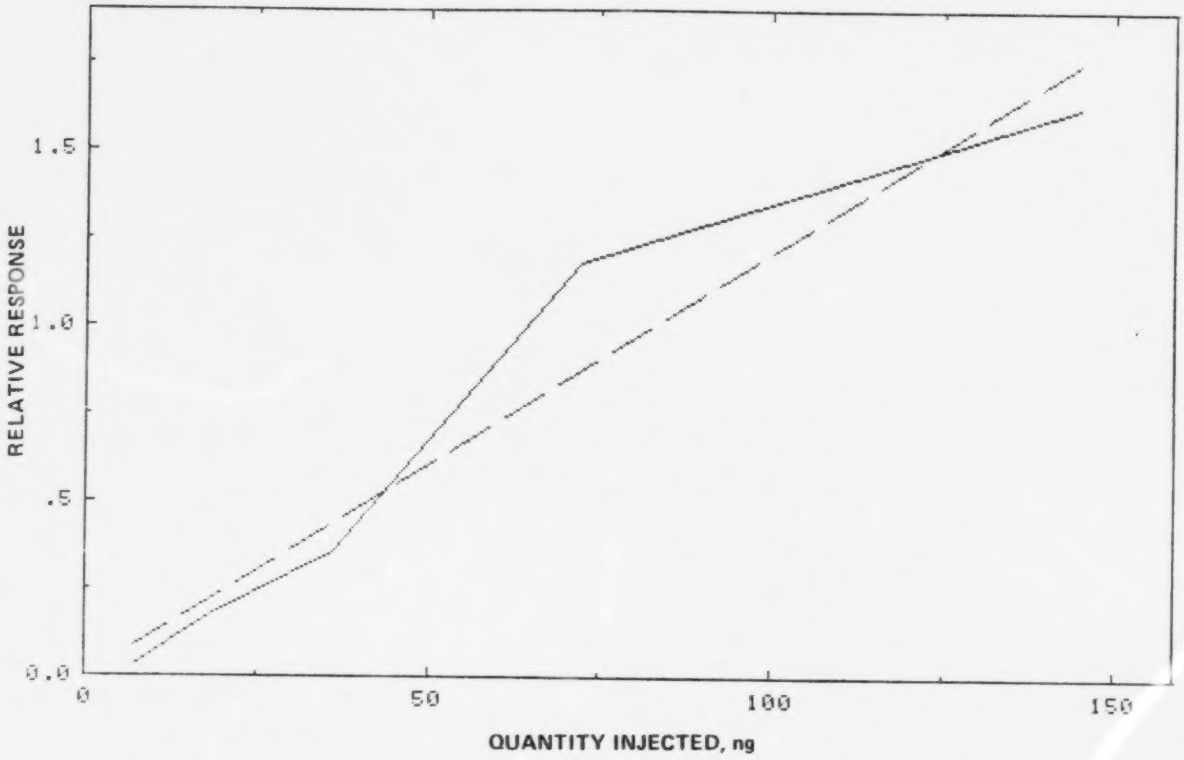


Figure D-118. Calibration curve for the determination of phosphorodithioic acid, O,O-dimethyl ester, S-ester with 3-(mercaptomethyl)-1,2,3-benzotriazin-4(3H)-one by GC/MS.

Range: 18 - 140 ng
Correlation coefficient: 0.969
Slope: 1.2×10^{-2}
y-Intercept: -1.1×10^{-4}

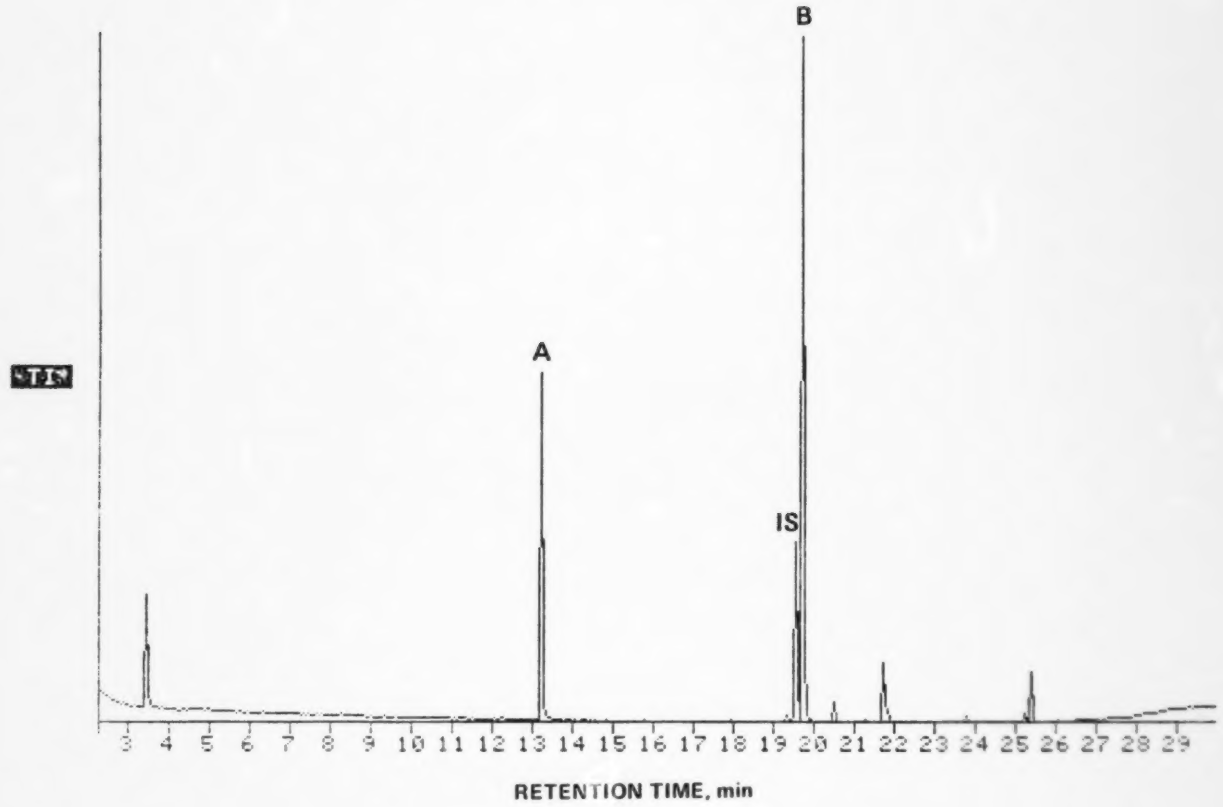
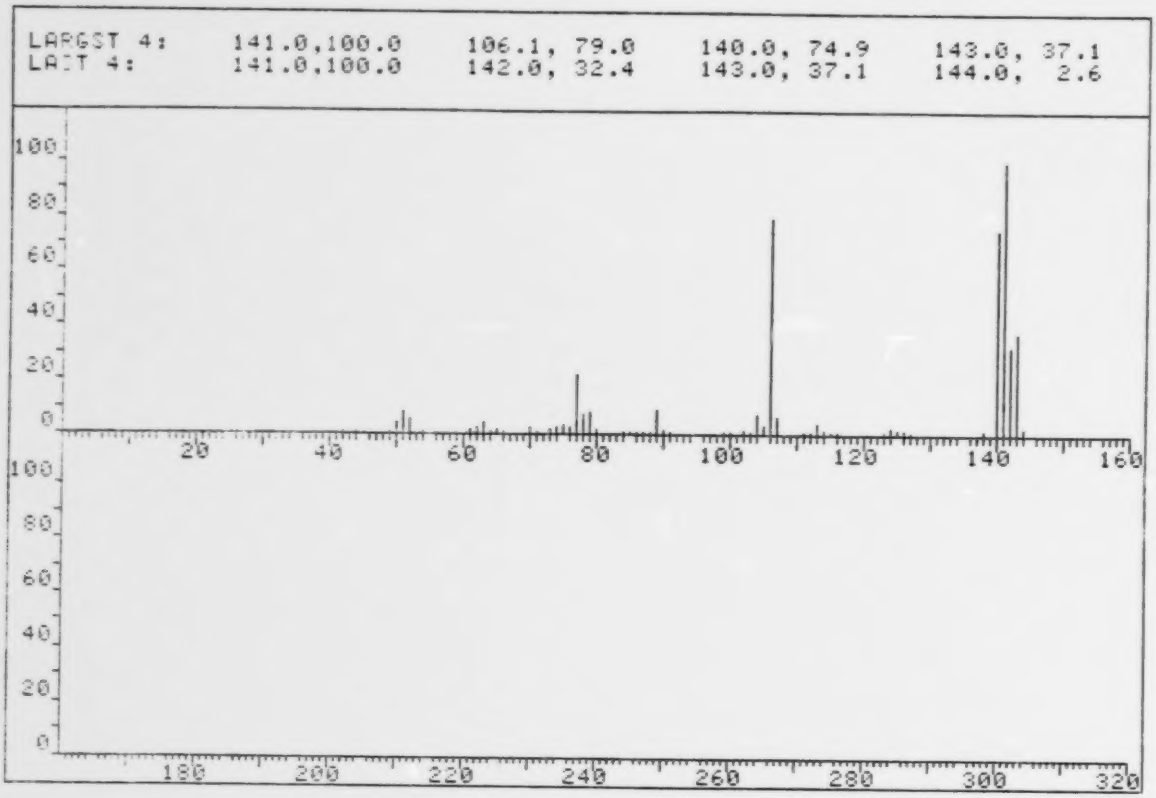


Figure D-119. Chromatogram by GC/MS for:

(A) o-toluidine, 5-chloro- (t_R = 13.2 min) and
(B) 1,4-naphthoquinone, 2,3-dichloro- (t_R = 19.8 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal



```
LARGST 4:     141.0,100.0     106.1, 79.0     140.0, 74.9     143.0, 37.1
LAST 4:       141.0,100.0     142.0, 32.4     143.0, 37.1     144.0,  2.6
```

Figure D-120. Mass spectrum for o-toluidine, 5-chloro-.

TABLE D-53. KEY IONS AND RELATIVE ABUNDANCES FOR o-TOLUIDINE, 5-CHLORO-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	1.0	71	1.0	90	1.4	115	1.3
42	1.1	73	2.0	91	.6	116	.6
49	.6	74	2.3	99	.9	123	.7
50	4.0	75	3.1	100	.8	124	2.4
51	7.6	76	2.6	102	1.3	125	1.6
52	5.9	77	21.6	103	.7	126	1.4
54	1.1	78	7.4	104	7.0	127	.6
61	1.5	79	7.7	105	2.8	138	1.5
62	2.1	80	1.6	106	79.0	140	74.9
63	4.5	85	.9	107	6.5	141	100.0
64	1.2	86	.7	111	.7	142	32.4
65	1.8	87	1.1	112	.9	143	37.1
66	1.1	88	.7	113	4.2	144	2.6
70	2.4	89	9.1	114	1.7		

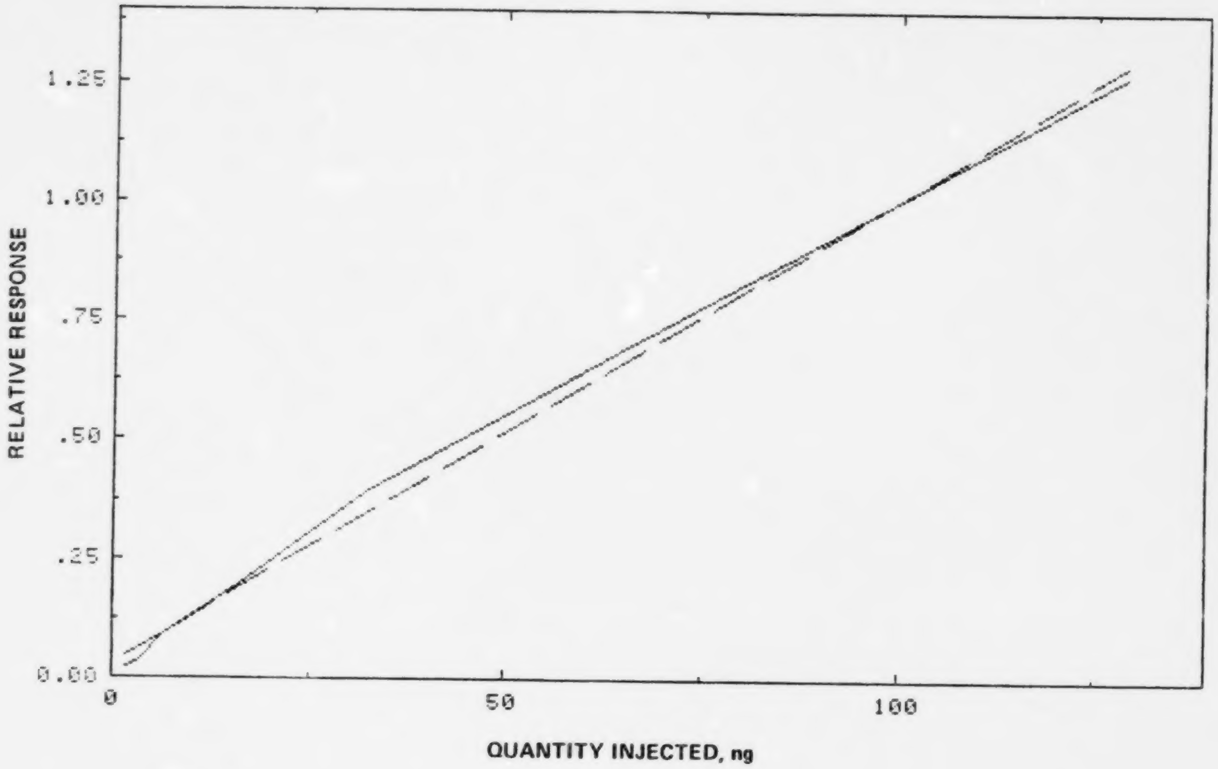
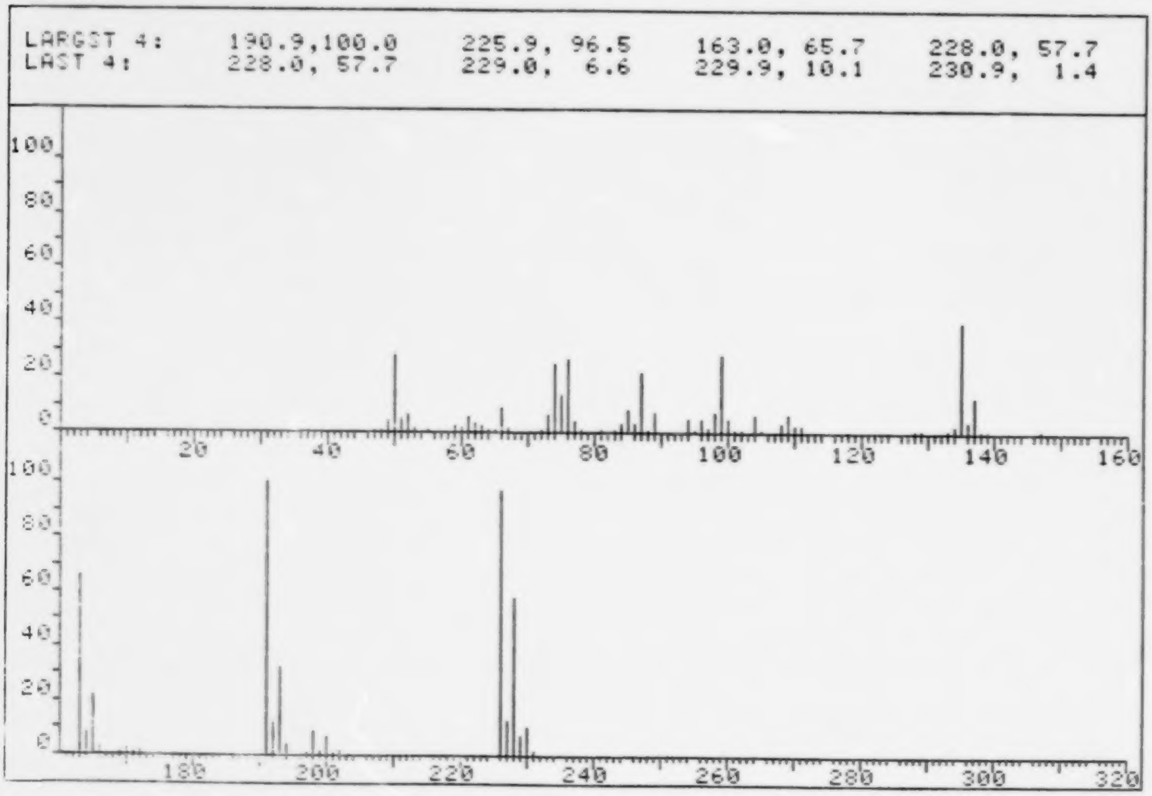


Figure D-121. Calibration curve for the determination of o-toluidine, 5-chloro- by GC/MS.

Range: 6.0 - 130 ng
Correlation coefficient: 0.998
Slope: 9.8×10^{-3}
y-Intercept: 3.0×10^{-2}



```
LARGST 4:     190.9,100.0      225.9, 96.5      163.0, 65.7      228.0, 57.7
LAST 4:       228.0, 57.7      229.0,  6.6      229.9, 10.1      230.9,  1.4
```

Figure D-122. **Mass spectrum for 1,4-naphthoquinone, 2,3-dichloro-.**

TABLE D-54. KEY IONS AND RELATIVE ABUNDANCES FOR 1,4-NAPHTHOQUINONE, 2,3-DICHLORO-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
47	1.2	75	13.8	104	6.2	170	2.2
48	.9	76	26.1	105	.8	171	.7
49	4.3	77	4.5	108	3.6	172	1.5
50	28.2	78	.8	109	6.1	191	100.0
51	5.1	81	.7	110	2.3	192	11.7
52	6.5	83	.5	111	2.8	193	32.2
53	1.9	84	2.8	128	.5	194	4.1
55	.8	85	7.7	129	.7	197	.7
59	2.3	86	3.6	133	1.1	198	8.7
60	1.9	87	21.8	134	2.4	199	1.2
61	5.7	88	1.0	135	40.2	200	6.5
62	3.3	89	7.0	136	4.1	201	.7
63	2.1	94	4.7	137	13.4	202	1.2
64	.7	95	1.0	138	1.2	226	96.5
65	.7	96	5.0	139	1.0	227	12.5
66	9.1	97	2.0	147	.6	228	57.7
67	1.3	98	7.2	163	65.7	229	6.6
67	1.3	99	28.0	164	7.6	230	10.1
72	1.1	100	4.8	165	21.5	231	1.4
73	6.5	101	1.3	166	2.1		
74	25.3	102	.5	169	.7		

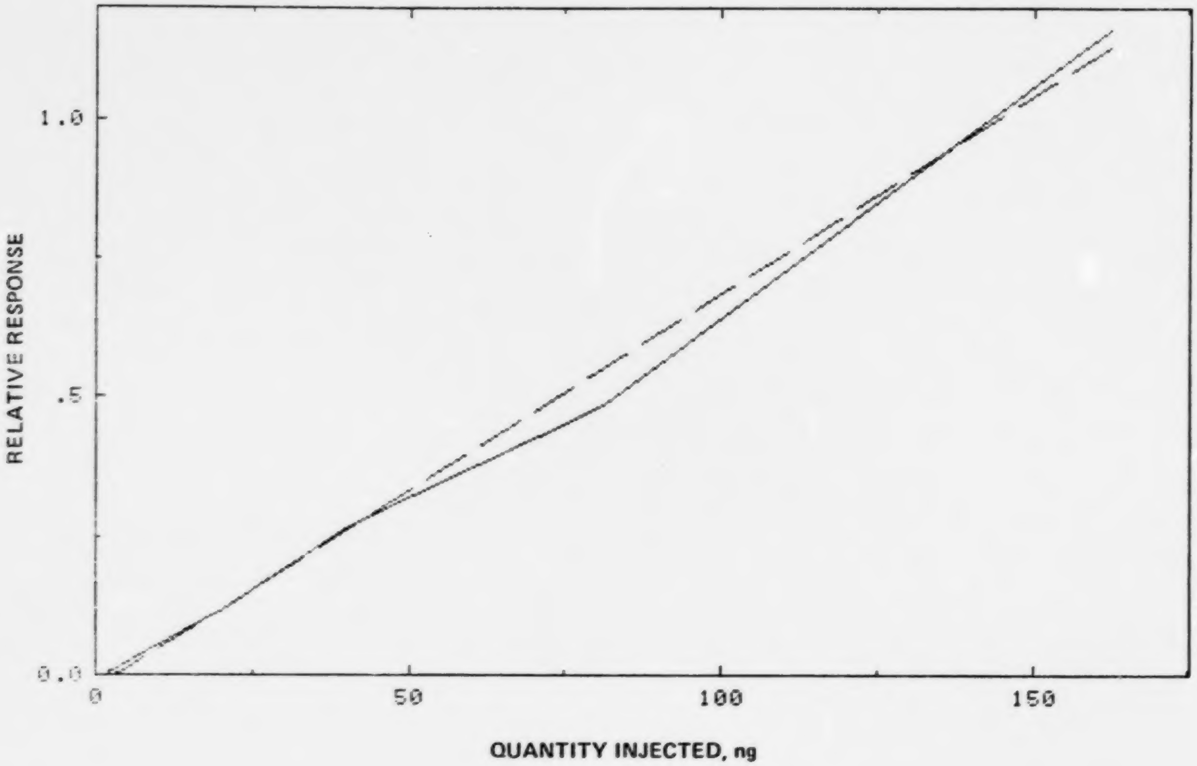


Figure D-123. Calibration curve for the determination of 1,4-naphthoquinone, 2,3-dichloro- by GC/MS.

Range: 20 - 160 ng
Correlation coefficient: 0.997
Slope: 7.1×10^{-3}
y-Intercept: -2.4×10^{-2}

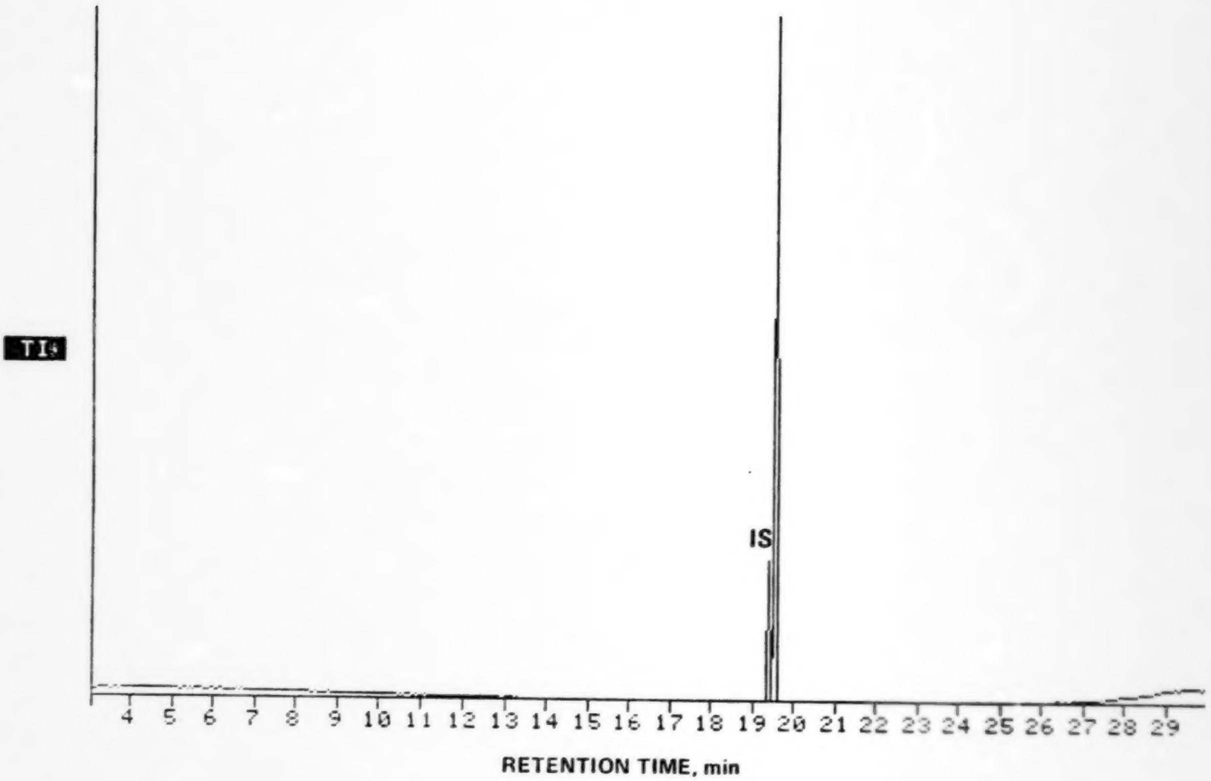


Figure D-124. Chromatogram by GC/MS for *p*-toluidine, *N*-(2-chloroethyl)-2,6-dinitro-*N*-propyl-*alpha*,*alpha*,*alpha*-trifluoro- (t_R = 19.5 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

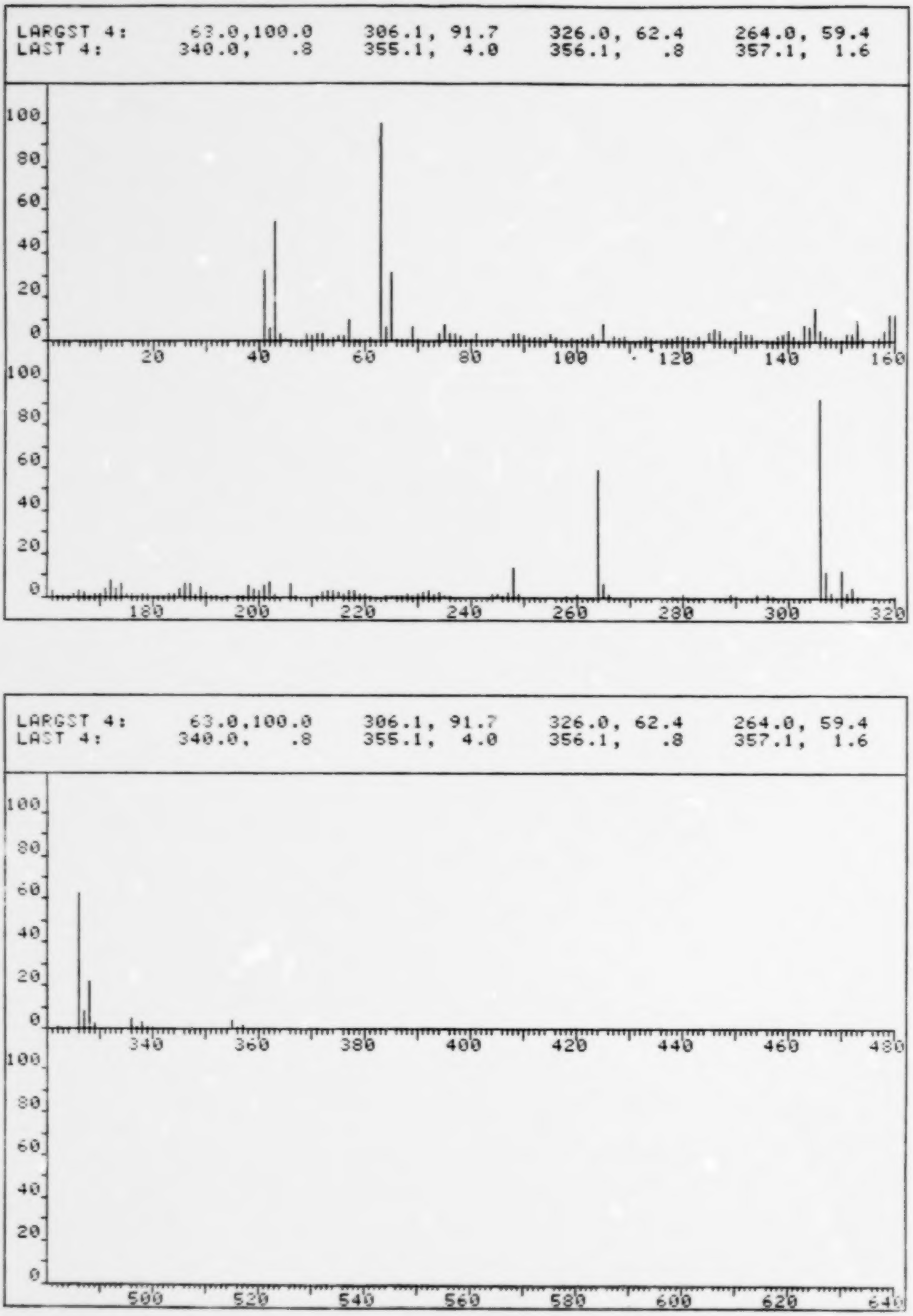


Figure D-125. Mass spectrum for p-toluidine, N-(2-chloroethyl)-2,6-dinitro-N-propyl-alpha,alpha,alpha-trifluoro-.

TABLE D-55. KEY IONS AND RELATIVE ABUNDANCES FOR *p*-TOLUIDINE, *N*-(2-CHLOROETHYL)-2,6-DINITRO-*N*-PROPYL-*ALPHA*,*ALPHA*,*ALPHA*-TRIFLUORO-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
41	32.5	102	1.8	147	2.6	213	2.5
42	5.7	103	3.0	148	2.0	214	2.5
43	54.8	104	1.2	151	3.1	215	1.9
44	3.0	105	7.9	152	3.1	216	1.2
49	3.7	106	1.0	153	9.9	217	2.6
50	2.2	107	2.2	154	1.8	218	2.5
51	3.7	108	1.6	157	1.5	219	1.3
52	3.7	109	2.2	158	5.2	220	1.2
54	1.8	112	1.0	159	12.1	228	1.3
55	2.3	113	2.5	160	12.3	231	1.9
56	2.5	114	1.9	161	3.1	232	3.4
57	10.0	115	1.3	165	1.1	233	1.1
58	1.1	116	1.0	166	2.5	234	1.7
61	1.4	117	1.7	167	1.8	244	1.0
63	100.0	118	1.4	169	1.1	245	1.1
64	6.2	119	2.2	170	1.1	247	2.0
65	31.9	120	2.3	171	3.7	248	13.5
66	1.0	121	1.3	172	7.4	249	1.5
69	6.4	123	2.6	173	4.0	260	1.1
70	1.0	125	4.0	174	6.2	264	59.4
74	2.8	126	6.1	175	1.6	265	6.0
75	7.1	127	4.7	176	1.6	280	1.3
76	3.8	128	1.6	179	1.3	289	1.3
77	3.5	129	1.1	183	1.1	294	1.1
78	2.6	130	1.9	184	1.6	296	1.0
81	3.1	131	5.0	185	3.8	306	91.7
82	1.0	132	3.3	186	6.2	307	11.7
83	1.2	133	3.0	187	5.8	308	1.7
88	2.8	134	1.2	188	1.6	310	12.9
89	3.5	135	1.1	189	4.9	311	2.0
90	2.4	137	1.1	190	1.7	312	4.4
91	1.7	138	2.3	198	5.1	326	62.4
92	1.5	139	2.9	199	3.7	327	8.6
93	1.6	140	4.6	200	2.7	328	21.5
95	3.6	141	2.4	201	5.6	329	2.7
96	1.5	143	7.1	202	6.5	336	5.2
99	1.4	144	6.7	203	1.3	338	3.5
100	1.3	145	15.6	206	6.3	355	4.0
101	1.3	146	4.8	212	2.0	357	1.6

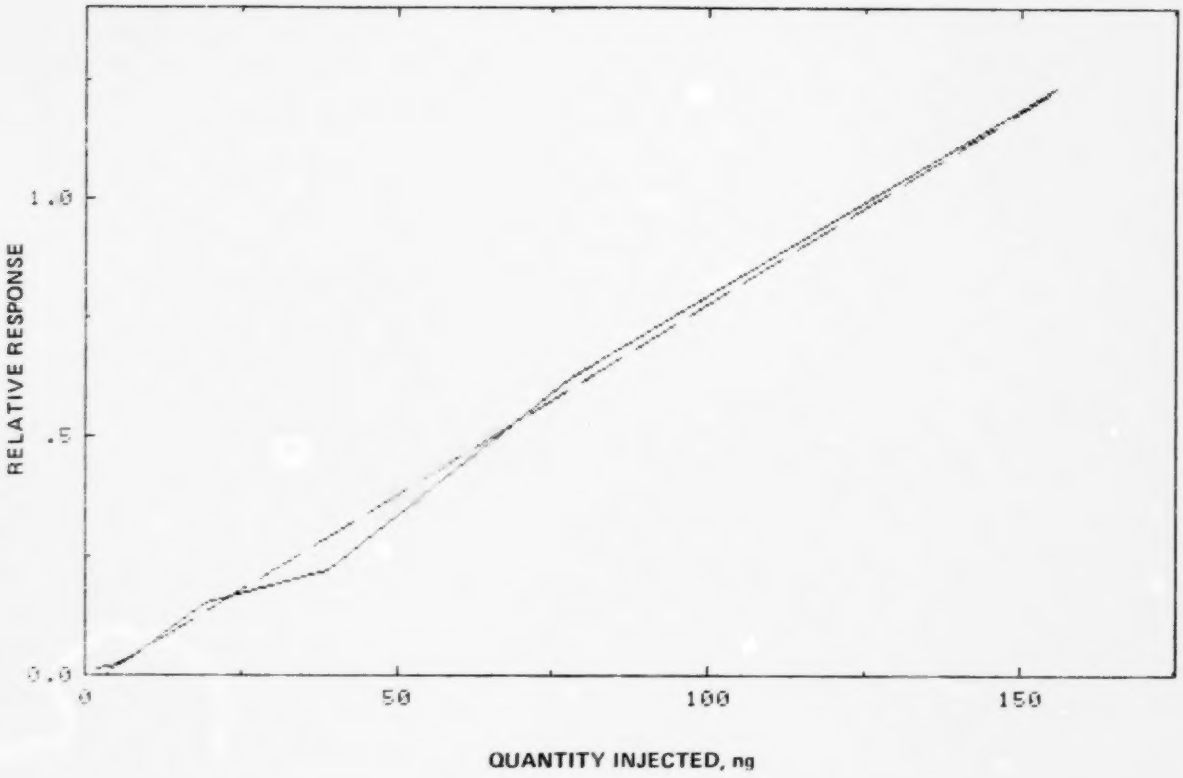


Figure D-126. Calibration curve for the determination of p-toluidine, N-(2-chloroethyl)-2,6-dinitro-N-propyl-alpha,alpha,alpha-trifluoro- by GC/MS.

Range: 7.8 - 160 ng
Correlation coefficient: 0.997
Slope: 8.0 x 10^{-3}
y-Intercept: -2.2 x 10^{-2}

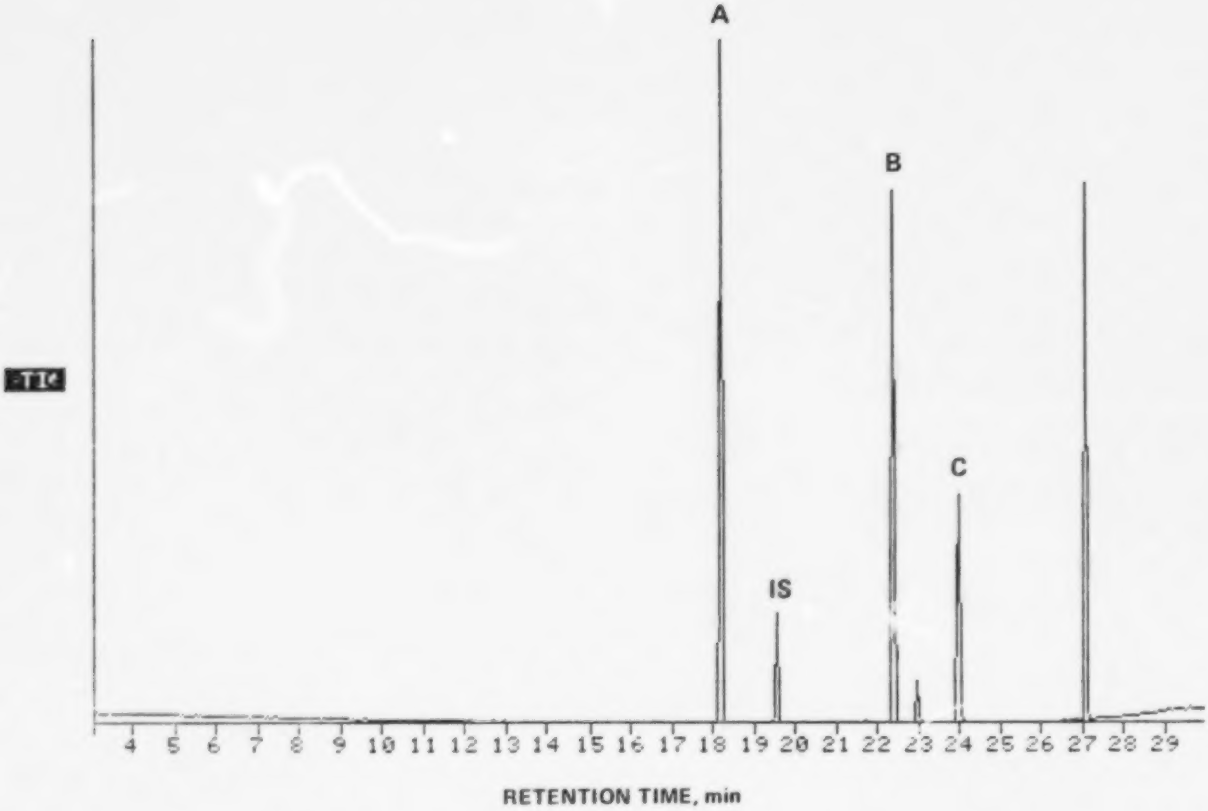


Figure D-127. Chromatogram by GC/MS for:

(A) p-toluidine, alpha,alpha,alpha-trifluoro-2,6-dinitro-N,N-dipropyl- (t_R = 18.1 min),
(B) heptachlor epoxide(t_R = 22.4 min), and
(C) ether, 2,4-dichlorophenyl p-nitrophenyl(t_R = 24.0 min).

Temperature program:
40 °C, 3 min isothermal
40 to 300 °C at 10 °C/min
300 °C, 10 min isothermal

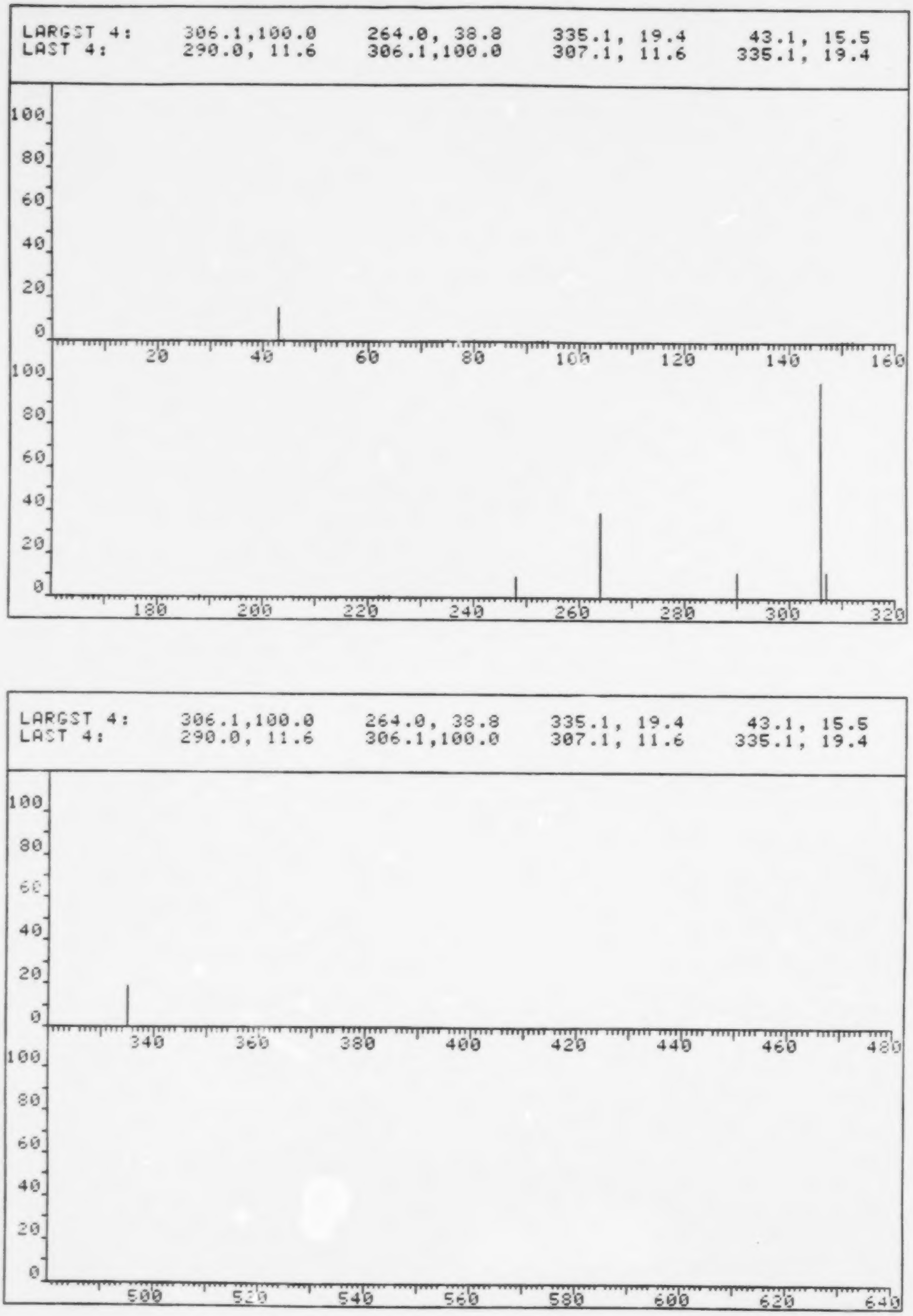


Figure D-128. Mass spectrum for *p*-toluidine, *alpha*,*alpha*,*alpha*-trifluoro-2,6-dinitro-*N*,*N*-dipropyl-.

TABLE D-56. KEY IONS AND RELATIVE ABUNDANCES FOR *p*-TOLUIDINE, *ALPHA*,*ALPHA*,*ALPHA*-TRIFLUORO-2,6-DINITRO-*N*,*N*-DIPROPYL-

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
43	15.5	248	9.7	290	11.6	307	11.6
44	1.0	264	38.8	306	100.0	335	19.4

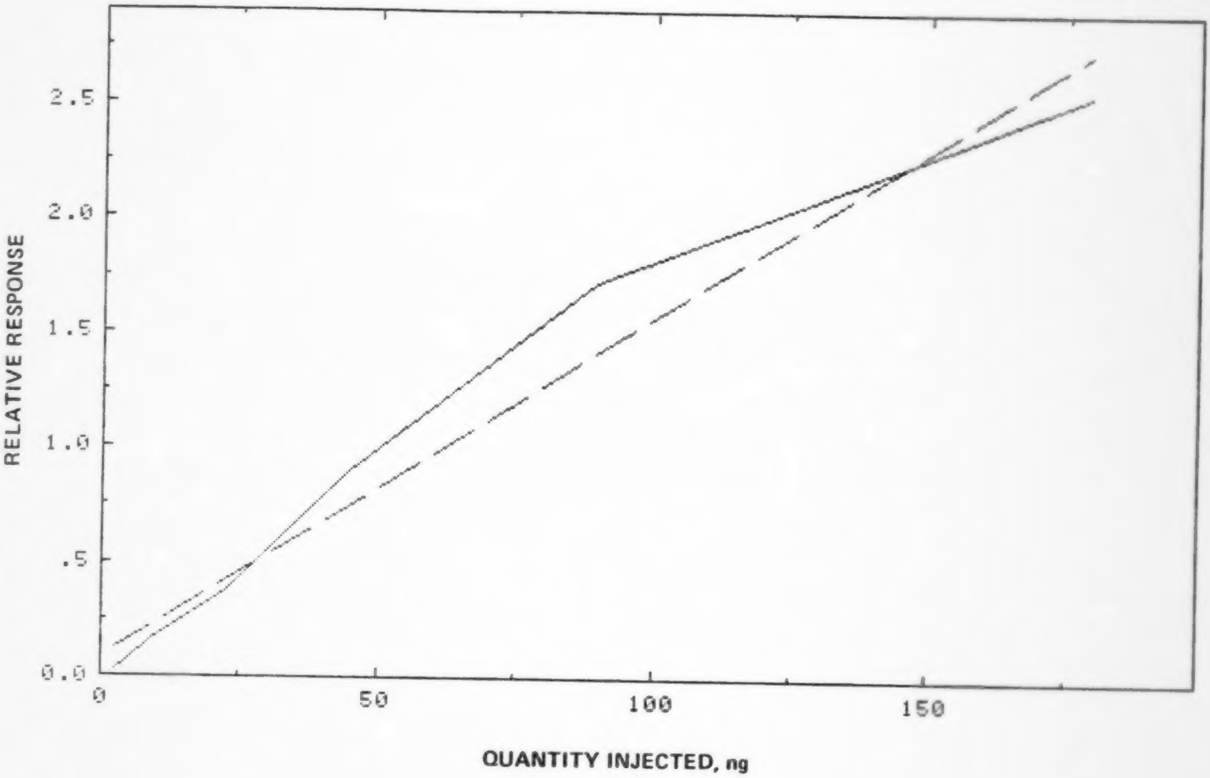


Figure D-129. Calibration curve for the determination of p-toluidine, alpha,alpha,alpha-trifluoro-2,6-dinitro-N,N-dipropyl- by GC/MS.

Range: 9.0 - 180 ng
Correlation coefficient: 0.985
Slope: 1.5 x 10^{-2}
y-Intercept: 8.7 x 10^{-2}

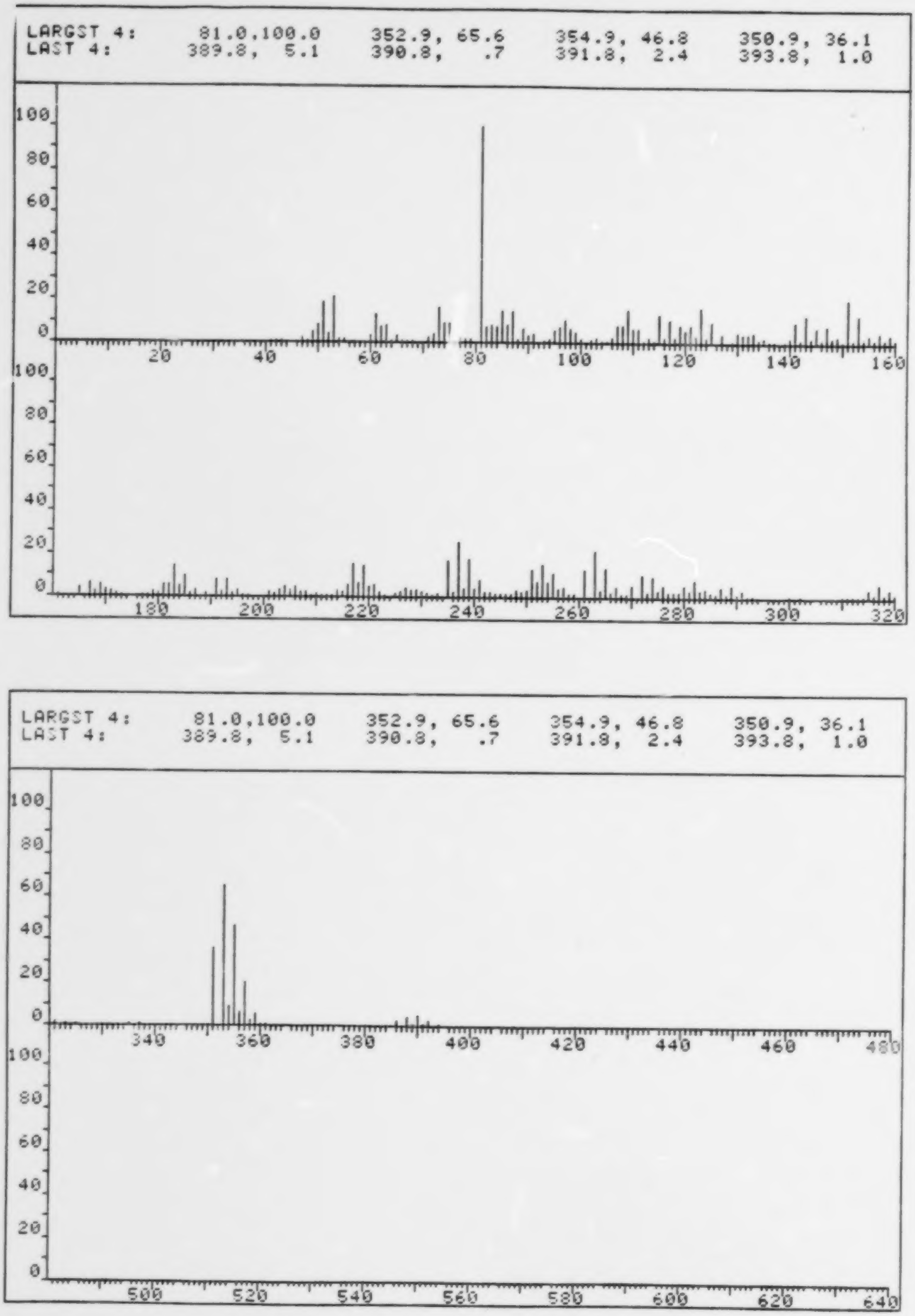


Figure D-130. Mass spectrum for heptachlor epoxide.

TABLE D-57. KEY IONS AND RELATIVE ABUNDANCES FOR HEPATACHLOR EPOXIDE

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
47	2.5	115	13.2	191	7.4	256	3.8
49	5.3	116	2.3	192	2.1	257	4.9
50	8.0	117	10.8	193	8.0	261	12.7
51	18.5	118	2.3	195	2.8	263	21.5
52	4.3	119	8.3	201	2.1	264	2.5
53	20.6	120	5.6	203	3.1	265	13.5
60	3.2	121	7.7	204	4.4	267	4.7
61	12.8	122	3.9	205	3.2	270	5.1
62	7.4	123	16.4	206	4.4	272	10.0
63	8.6	124	2.6	207	2.3	273	2.0
65	2.8	125	9.8	214	3.1	274	9.2
71	2.1	127	4.2	215	2.1	275	2.4
72	4.6	130	4.9	216	5.7	276	5.1
73	16.1	131	4.2	217	15.0	279	2.0
74	9.4	132	4.4	218	5.8	280	5.5
75	9.0	133	5.2	219	13.9	281	2.7
77	2.7	135	2.4	220	4.4	282	7.4
81	100.0	140	2.1	221	5.4	283	3.1
82	7.2	141	10.1	222	2.2	284	3.6
83	8.4	143	13.0	226	2.3	285	2.2
84	7.3	144	2.6	227	3.6	287	4.3
85	14.6	145	7.0	228	2.7	289	5.3
86	8.7	147	7.8	229	3.3	291	3.1
87	15.0	148	2.1	235	16.3	315	4.2
89	6.5	149	3.1	236	2.1	317	6.2
90	2.9	151	20.4	237	25.4	319	4.1
91	4.1	153	13.0	238	3.9	351	36.1
95	5.5	155	4.6	239	17.7	353	65.6
96	7.2	157	5.1	240	4.1	354	9.3
97	10.2	159	4.4	241	7.9	355	46.8
98	6.6	165	3.7	242	2.0	356	6.5
99	5.0	167	6.0	243	2.3	357	20.1
103	2.2	169	5.1	248	3.1	358	2.4
106	2.7	170	2.9	249	2.3	359	5.4
107	8.7	181	5.7	250	3.3	386	2.6
108	8.7	182	5.7	251	12.6	388	4.4
109	15.4	183	13.9	252	6.8	390	5.1
110	6.2	184	4.3	253	15.1	392	2.4
111	6.4	185	9.6	254	7.0		
113	2.4	187	2.9	255	10.6		

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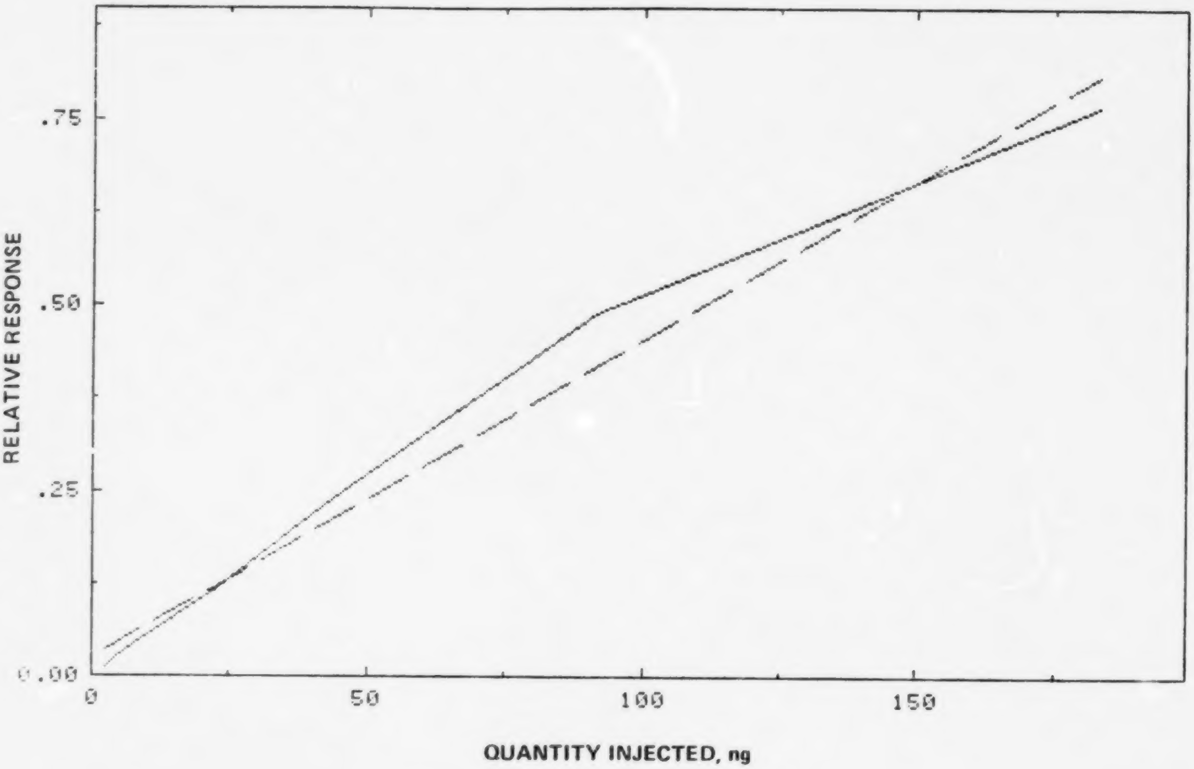
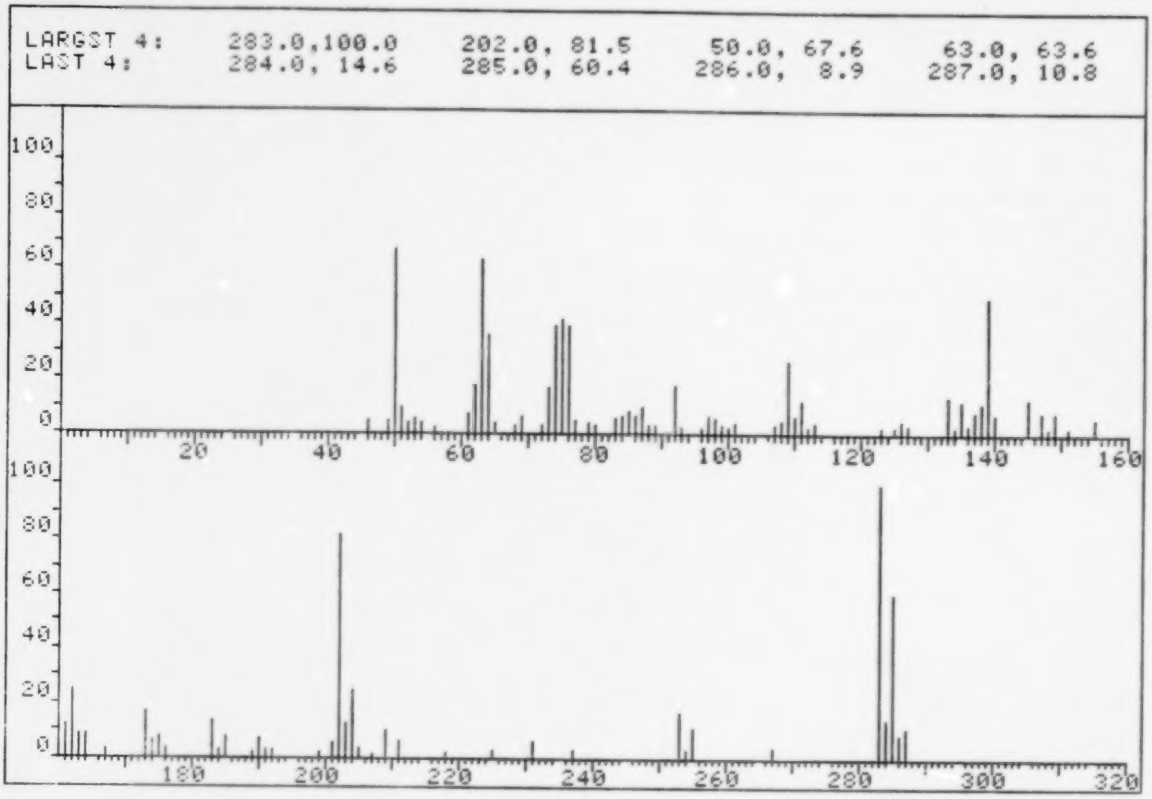


Figure D-131. Calibration curve for the determination of heptachlor epoxide by GC/MS.

Range: 9.2 - 180 ng
Correlation coefficient: 0.991
Slope: 4.3×10^{-3}
y-Intercept: 2.7×10^{-2}



```
LARGST 4:    283.0,100.0    202.0, 81.5     50.0, 67.6     63.0, 63.6
LAST 4:      284.0, 14.6    285.0, 60.4    286.0,  8.9    287.0, 10.8
```

Figure D-132. Mass spectrum for ether, 2,4-dichlorophenyl p-nitrophenyl.

TABLE D-58. KEY IONS AND RELATIVE ABUNDANCES FOR ETHER, 2,4-DICHLOROPHENYL p-NITROPHENYL

M/Z	Abundance	M/Z	Abundance	M/Z	Abundance	M/Z	Abundance
46	4.8	86	6.2	135	12.0	191	2.6
49	5.0	87	9.8	136	3.1	192	2.6
50	67.6	88	3.1	137	8.4	199	2.4
51	9.6	89	3.6	138	11.3	201	5.3
52	4.3	92	18.0	139	49.9	202	81.5
53	5.8	93	2.6	140	7.0	203	12.5
54	4.3	96	2.4	145	12.7	204	24.9
56	2.4	97	6.5	147	8.6	205	4.1
61	7.4	98	5.8	148	2.6	207	1.0
62	18.0	99	3.8	149	8.6	209	9.8
63	63.5	100	2.6	151	2.6	211	6.0
64	36.2	101	4.3	155	5.8	218	2.4
65	4.6	107	3.1	161	11.5	225	2.6
68	2.9	108	5.0	162	24.7	231	6.5
69	6.7	109	26.1	163	8.9	237	3.1
72	3.8	110	6.2	164	8.9	253	16.8
73	17.3	111	12.0	167	3.1	254	2.9
74	39.1	112	2.6	173	16.8	255	10.8
75	42.2	113	4.1	174	6.5	267	3.8
76	39.3	123	2.4	175	7.7	283	100.0
77	4.8	123	2.4	176	3.6	284	14.6
79	4.3	125	2.6	183	13.2	285	60.4
80	3.1	126	4.8	184	2.6	286	8.9
83	5.5	127	3.6	185	7.7	287	10.8
84	6.7	133	13.9	189	2.4		
85	7.7	134	2.4	190	7.2		

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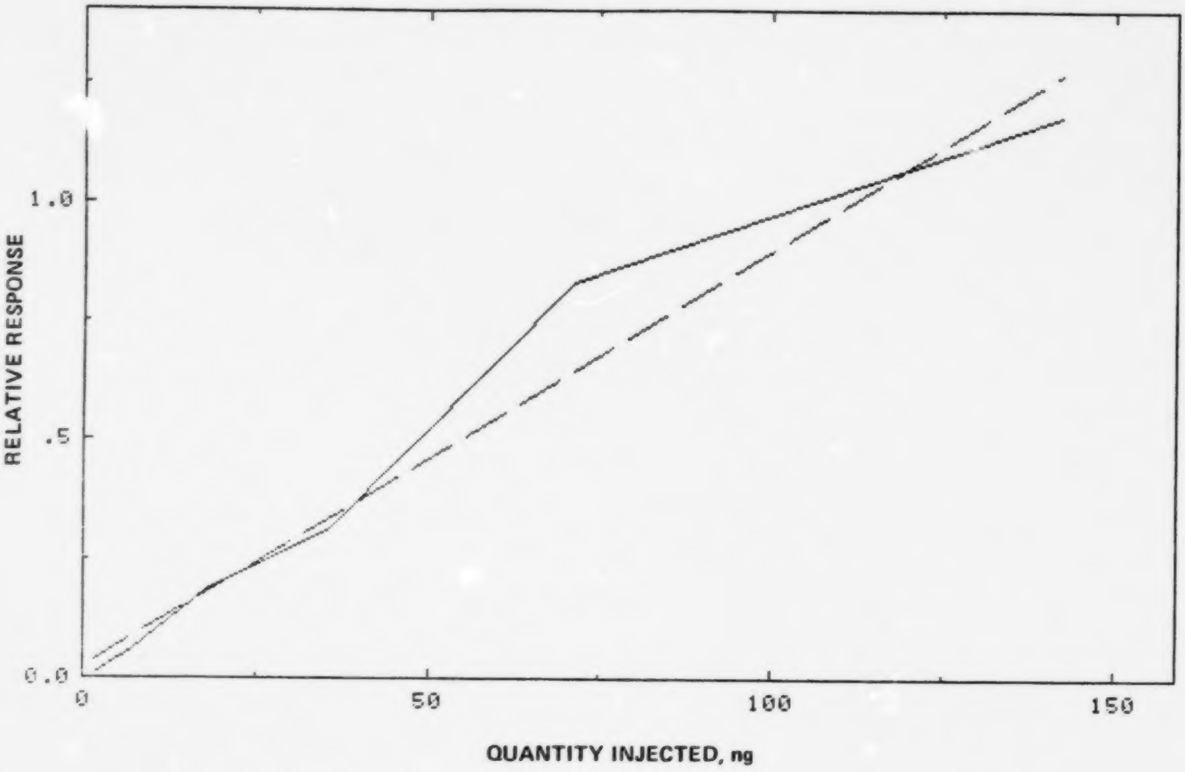

Figure D-133. Calibration curve for the determination of ether, 2,4-dichlorophenyl p-nitrophenyl by GC/MS.

Range: 18 - 140 ng
Correlation coefficient: 0.982
Slope: 8.7×10^{-3}
y-Intercept: 2.3×10^{-2}